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Title: FEASIBILITY STUDIES FOR COMPOSTING PAPER MILL WASTE

PROJECT ADMINISTRATION DATA

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Project Director TORNABENE, THOMAS

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FINAL REPORT

TITLE:	Feasability studies for composting paper mill waste
SPONSOR:	Weyerhaeuser
CONTRACT NO.	FR37270-DJS1
DURATION:	June 1, 1996 to October 31, 1997
PI:	Thomas G. Tornabene Georgia Institute of Technology
Co-PI:	K. C. Das University of Georgia
DATE OF FINAL REPORT:	October, 1997

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ABSTRACT

Paper mill waste generated at the Weyerhaeuser, Flint River, plant can be effectively composted under controlled conditions. The mix of paper mill waste requires a moisture content of 40-55%, a pH below 7.5 to allow for maximum mold formation, relatively fresh primary sludge from the primary clarifier which provides active biological cultures, a mixture content with sufficient bulking to allow for air filtration, and a supplement of nitrogen compounds. KNO_3 and NH_4NO_3 were the most effective supplements for composting the waste with the least effective being Urea. The addition of other supplements such as Chicken Litter, $NH_4H_2PO_4$, and NH_4Cl and combinations were less effective. The additions of mineral salts and commercial additives advertised or reported to stimulate composting were ineffective in this study. It was concluded from the bench-top experiments that under suitable conditions 20% of the paper mill waste can be mineralized within a 30 day cycle.

The potential for anaerobically converting paper mill waste to CH_4 and $C0_2$ was demonstrated with as much as 60% of the total raw material mineralized. The supplements required for this conversion in a 40-60 day cycle was NH_4NO_3 or NH_4Cl or a combination of KNO_3 and NH_4Cl in sequence. A starter culture was essential for rapid start-up. $NH_4H_2PO_4$, KNO_3, Urea or Chicken Litter supplement stimulated CO_2 production with relatively small quantities of CH_4. The addition of NH_4SO_4 resulted in sulfur being reduced. N_2O and traces of NO were observed in all systems containing NO_3^- as a supplement.

FINAL REPORT

A. BENCH TOP FEASIBILITY STUDY

INTRODUCTION

The thrust of the research study had two goals. One of them was to reduce the quantity of waste products accumulated daily at the Flint River site. The other was to test the feasibility of converting waste into a product that could be returned to the earth as a soil amender. The principal method to be utilized was traditional composting. The principal target was to measure the degree of mineralization that could occur in a reasonable time period and to evaluate the quality of the product.

During the study, it was decided that anaerobic methods should also be tested for the potential for mineralization of the wood waste as well as for methane gas production. These experiments were initiated six months after the start of the program.

MATERIALS AND METHODS

Materials: The waste (raw) materials available for this study are summarized in Appendix 1. Screening Knots were included in pilot studies but are no longer a major waste product at the Plant and therefore was excluded. Fly Ash was included but decidedly limited in its applicability for supporting microbial growth because of high pH values of around 12. Also, there was the concern of too high of an ash content causing metal shock to microbial cells. Therefore, boiler fly ash was used as a 1% enrichment and usually required pH adjustment with acid prior to its addition. Lime mud was not used in the study because of it high pH property and the nature of its composition that would be detrimental to microbial activity unless greatly diluted.

Primary sludge, bark and grit were chosen as the test materials because of its high organic carbon content. Primary sludge contained significant quantities of sand. Depending on the lot of Primary sludge collected at the Flint River site, the sand content varied significantly; accordingly, the ash contents recorded in the results section reflects this variation. The bark and grit were used as both the bulking agents and reactant but with the primary sludge representing the principal reactant. The volume ratios of the principal reactants used in preparing the mixtures were determined volumetrically in 1 and 5 gallon buckets and then weighed. All other additives were measured gravimetrically.

Since the starting materials are largely woody in nature (cellulosic, hemicellulosic), the nitrogen contents were virtually nonexistent. The nitrogen supplements used in the study were chicken litter from chicken ranches in the Flint River vicinity, NH_4NO_3, NH_4Cl, $NH_4H_2PO_4$, Urea, KNO_3, NH_4SO_4 or combinations thereof. Although the starting materials were relatively high in inorganics (Appendix 2), complex mineral salts commonly used to support microbial growth was tested as supplements. The Mineral Media consisted of KH_2PO_4 - 4.5g; Na_2HPO_4 - 3g; KNO_3 - 5g; $MgSO_4$ - 0.25g; H_3BO_3 - 2.9g; $MnCl_2$-4aq - 1.8mg; $ZnSO_4$-7aq - 0.22mg; $CuSO_4$-5aq - 0.08mg; H_2MoO_4-1aq - 0.015mg; and $FeCl_3$ - 0.0025mg per L volume of reactants.

Reactor designs: The aerobic reactors were made of clear plexiglass configured rectangularly with a removal lid that was bolted to the assemblage to create a water and air tight reactors. The reactors were equipped with sampling ports, air injection system, exhaust ports, air flow rate meters and temperature probes. Two sizes were used in this study. The largest had a sixty-liter capacity (18 cm x 18 cm x 153 cm) and the others were of 20 liter capacities (18 cm x 18 cm x 65 cm). Each reactor contained a metal grid positioned 10 cm above the base plate to create a plenum for air delivery in an upward flow through the reactor. The aeration rate was maintained between 5 - 7.2 m^3/lb/day of compost mix. Since the air flow fluctuated and the volume of the reactant reduced in volumne relative to biological activity, the quantity of CO_2 produced in each reactor was normalized by multiplying the amount of CO_2 in the gas sample by 0.1 of the flow rate volume.

The anaerobic reactors consisted of 2, 4 and 6 L wide-mouth Erlemeyer-type flasks and 3 L Fernbach flasks with stoppers containing a septum for gas sampling and ports for connection to an open-ended Hg tube for recording gas pressures and for the daily release of the gas pressures and/ or a graded inverted cyclinder filled with water to measure gas displacement of the water in the cylinder. Each of the reactor vessels contained water with the compost mix in a slurry state. Two of the 20 L reactors used in the aerobic study were also employed in the anaerobic study but as a solid phase. In these modes, the inflow and outflow air ports were plugged with serological septa. In some cases, the reactors were filled and sealed whereas in others they were flushed with N_2 before plugging the ports.

Inoculation: No extrinsic inocula were used. All microbial activity was by in situ cultures. For the anaerobic studies, however, the standard mix of reactants were slurrried without nitrogen additive and incubated at room temperature. These slurries were used as seed cultures. The ideal seed cultures are those obtained from the vessels actively producing CO_2 and CH_4; however, the contents of those reactors were used to quantify the percentages of the bioconversion processes, and were not available as seed cultures.

Microbial Counts: Enumerations were determined by modified method described by Craft and Nelson (Appl. Environ. Microbiol. 62:1550-1557(1996)). Enumerations for bacteria, fungi and actinomycetes from the compost mixtures were done by taking 1g of compost and suspending it in 99 mL of 0.1% water agar (1g agar/L). The suspensions were then blended for 45s in a Waring blender. Tenfold dilutions were prepared with sterile distilled water and 0.1 ml of the suspensions spread on 4 replicate plates of the following media:

- for fungi - 0.3X potato dextrose agar with 50 μg/ml each of streptomycin and ampicillin.
- for bacteria and actinomycetes - 1/10 & 1/50-strength trypticase soy agar, respectively.

All plates were incubated for 72 hrs at 24 C. Colonies were enumerated and populations were expressed as CFU (colony forming units) per gram of compost.

Microbial Cultivations: Molds from the aerobic reactors were transferred by inoculating loops to agar plates containing 1% cellulose or cellobiose. The plates were then incubated at 24°C for up to 2 weeks. The plates were evaluated as positive or negative for supporting growth.

Analytical Procedure: Nitrogen was determined by the standard Kjeldahl method and for CHN by combustion analyses. The total volatile solids (TVS), ash and moisture contents were determined by drying the samples overnight in an oven at 100 C, followed by ashing in a muffle oven at 600 C for 45 minutes with the differences in weights recorded gravimetrically. No chemical marker analyses were performed to attempt to determine the amount of microbial biomass that may have accumulated during the bioconversion process and which would contribute to the TVS value. Cautions were taken to not overly shake or mix the dried compost samples during this procedure since the sand readily separated from the dried particles and filtered to the bottom of the container (it stuck firmly to wet particles), otherwise making it impossible to obtain consistent measurements. Even with care in sampling and in selecting samples, representative samples were difficult to obtain because of the diverse nature of the starting materials being fibrous clumps (primary sludge) and a wide range in sizes of bark and grit chips. Even with a ball-mill grinder, representative samples were flawed by the separation of the sand from the particles.

For quntifiying the bioconversion rates in the anaerobic reactors, the total volume was removed from the reactor at the end of the run and dried in an oven and gravimetrically measured. Gas analyses were performed with a HP 5890A Gas Chromatograph equipped with a thermoconductivity detector and a 3 m x 0.6 cm SS column packed with 100/120 carbosieve S-II. Identities of the gases were determined by comparing RT values to those of authentic standards of H_2, CO_2, CH_4, CO, N_20, NO, O_2, H_2O, SO_2, and NH_3. The methods used to calculate the volume of gases emitted from the anaerobic reactors was accomplished by Hg pressure gauges according to standard gas laws (Appendix 3) followed by measuring the amount of gas displacement of acidified water in a graded inverted cylinders. The error from CO_2 solubility in water was estimated to be only 1%. The pH of the samples was determined by placing the compost mix into a beaker with a minimal amount of distilled water required to suspend the mix. Samples were recorded directly after a 30 min soaking period as well as by measuring the pH of its filtrate.

RESULTS

Aerobic Compost Reactors: The results from the first three reactors assembled in this study are not included in the tables and figures in this final report and are only described in this paragraph. The reactor mixtures were the same as those described in Table 1 (reactor 1) but with no pH adjustments or additives. The pH values at the start (t_o) were 8.5 - 9.2. After fourteen days of aeration, no CO_2 or temperature changes were observed and they were discontinued.

The compositions of the mixtures used in the composting bioconversion studies are summarized in Table 1. The reactor numbers in Table 1 correspond to the reactor numbers as well as the figure numbers used throughout the text. Accordingly, the CO_2 emission profiles that describe the relative biological activities of each of the compost reactors over the course of their operations are listed as Reactors 1-26. The moisture, pH, ash and TVS values for each reactor operated with forced-air in a compost mode are summarized in Table 2. The data in Table 2 and the CO_2 profiles in Reactors 1-26 may not directly correspond since the TVSs include the total solid fraction (biomass, fermentation products, etc.) accumulated in the reactor.

Reactors 1-3 (Table 1) differ from each other particularly in terms of the ratio and contents of the compost substrates, the method of pH adjustment and that Urea as 1% of the mix was added to reactor 2 that lacked an addition of fly-ash. The reason they are linked together is because of the low percentage of CO_2 yields, indicating low biological activity. The use of Urea as a nitrogen source in composting was repeated with similar results as well as used later as a mixture with other nitrogen sources (Reactor 15 to be discussed later). The results from Reactor 1-3 are similar in that the adjustment of pH clearly showed improved biological activity but the increase in CO_2 was still judged as too low for expected productive bioconversion rates. The effect of pH adjustment is most evident in Reactor 1 after pH was adjusted after 10 days of operation. Generally, no pH adjustments were necessary if the concentration of fly-ash was kept below 0.1% of the composition. Nonetheless, fly-ash was not used in further mixes unless adjusted to near neutrality prior to its addition to the compost mix. The addition of Urea (Reactor 2) had relatively little if any effect on the compost mix. This result was attributed to the oligotrophic environment that exist, or to the location of the various nutrients in the mix that are not easily reached by the microorganisms. While the basic data summarizing the reactions of Reactors 1-3 show only 1.7 - 3.6% conversion of the compost to humic type material (Table 2) within 16 days, the volume of the compost mix in the reactor reduced some 30%. This volume reduction was seen in all reactors in which biological activity occurred. This reduction in volume was attributed to biological alterations of components of the mix and to biological activities in general. It was not attributed to settling and compaction over-time due to gravity because reactors without biological activities did not reduce in volume under the same environmental conditions and incubation times. Later on it will be noted that highly biological reactions gave reduction rates approaching 40%.

Reactors 4-7 are compared because each of them was constructed with the same mix and fortified with NH_4NO_3. Reactor 5 contained twice the amount of salt as the other three (Table 1). Reactors 4 and 5 contained no additional supplements whereas Reactor 6 received 200 mL of a product advertized as an effective enhancer of composting, sold as Ecosystem Plus by Neozyme Inc., Newport Beach, CA 92663. Although the exact content of Neozyme was not made public, it is clearly a Vinasse solution (yeast fermentation broth) enriched with a surfactant (soap). Assumably, the vinasse is rich in B-vitamins, salts and fermentation by-products that are good supplements to most media for growing microorganisms. Further, the surfactant should increase the potential for interaction between the substrate and microorganisms. Reactor 7 contained "Formula # 615 - special formulation for composting". This product was sold by Deutrel Laboratories, Inc., Palmdale, CA. Warning label states that it contains H_2SO_4 (the product had a pH of 1 and appeared to contain little more than trace mineral salts). Reactors 4-7 (and others thereafter) were particularly distinguished by white mycelium of the fungi that completely engulfed the compost mix, giving the appearance of snow. The mold appeared around the 12th day and lasted to the 27-30th day. The appearance of the white mold was consistently observed in all of the additional reactors receiving supplements of NH_4NO_3 as the only ammonium salt. Other notable features were the increases in temperature to 35 - 40°C corresponding to CO_2 emission levels of 15 to 62% of the effluent gases (figures of Reactors 4-7). Temperatures were not expected to get higher because of the relatively large surface area of the reactors that were not insulated. The highest CO_2 levels were recorded for Reactor 6 which contained Neozyme supplement. This increase was attributed to microbial activity feeding off of the Neozyme products since the TVS and ash contents (Table 2) were not significantly different. In fact, the highest bioconversion of substrates was observed in Reactor 5 which contained 100 gm of NH_4NO_3. The CO_2 effluent levels were not the highest recorded for this set but they remained at an elevated level for a longer time period (figures of Reactors 4-7). In fact, the CO_2 profiles demonstrate a possible diauxic curve for those containing NH_4NO_3; the NH_4^+ of NH_4NO_3 is perhaps used first with the NO_3^- utilization occurring afterwards. Thus, a demonstration of two different culture enrichments. The basic conclusions were that neither Ecosystem Plus nor Formula #615 were effective in enhancing composting and that NH_4NO_3 had a significant impact on bioconversion rates.

In pursuit of demonstrating the importance of inorganic nitrogen, Reactors 8-12 were assembled to measure the concentration range of NH_4NO_3 that affected the bioconversion rate. The amounts of NH_4NO_3 added ranged from 30 g to 120 g (Table 1). The results are not as clear as one would expect. This was attributed, in part, to the difficulty in solubilizing more than 60 g of the salt into a minimal amount of H_2O to be sprayed onto the compost mix while keeping the moisture content of the compost mix below 55%. Moisture contents more than 55%, with the added H_2O coming from the metabolic activity, resulted in nonuniformed air diffusion through the matrix. The data in Table 2 indicate that the poorest conversion rate occurred in Reactor 8 when the C:N content was about 123:1. The bioconversion results on the basis of TVS was only 2.6%, similar to that for Reactor 7 with higher NH_4NO_3 plus Formula #615. The best conversion rate of 7.9% (based on TVS) occurred in Reactor 11 with a C:N ratio of 43:1. Yet the CO_2 profile of Reactor 11 (operated for 30 days) showed less CO_2 output than reactor 9 which had a C:N of 77:1 and a TVS conversion of 6.0%. The amount of gas recorded daily over the life of the reactor does not proportionately reflect the differences in the bioconversion rates (TVS) reported in Table 2. Reactor 12 with the C:N ratio of 30:1 was comparable in CO_2 profiles and TVS conversion rates to Reactor 9 (C:N of 77:1). In summary, it appears that the highest bioconversion rate expected when only NH_4NO_3 is added to the raw compost mix is 8% (Reactor 11, Tables 1 & 2).

To further test the effects of inorganic nitrogen on composting of paper mill waste, Reactors 13-16 were assembled (Table 1), each containing a different nitrogen salt. Reactor 13 contained NH_4Cl, Reactor 14 contained $NH_4H_2PO_4$ and Reactor 15 contained both of the previous salts plus NH_4NO_3 and Urea (Table 1). All of the salts were added to give a calculated final concentration of C:N of 30:1. The gas flow emission profiles (figures of Reactors 13-15) and final TVSs (Table 2) indicate that the more diverse the nitrogen source the more simultaneous (rather than succession) enrichment of the diverse population of microorganisms which exist in the compost mix. This conclusion was based on the relatively high gas evolution that occurred in Reactor 15. The data is inconclusive as to the preferred nitrogen source with the exception that when Urea was added separately (Reactor 2, Table 2) a positive effect was noted but with relatively small bioconversion rates.

One of the limitations of the compost reactor is the localization of nutrient and microbial events because of the solid nature of the composition and absence of a diffusion medium. Therefore, Reactor 16 (Table 1) was assembled but in a "pond-like" configuration, based on the rationale that nutrients and microorganisms could be mixed and distributed throughout the matrix. An aliquot of the compost mix used to construct Reactor 9 (Table 1) was suspended in 4 Liters of H_2O and aerated. The percentages of CO_2 in the effluent air (figure of Reactor 16) was almost at background levels and, if present, diluted in the effluent air stream. After 43 days there was no reactivity that was measurable in Reactor 16. It was terminated with the conclusion that the aerated system (lagoon style) was either ineffective in biodegradation of the pulp and fiber, with the system employed for the test, or all of the CO_2 dissolved in the water. A total weight determination was not none, thus no decision can be made about the results.

Another test was conducted with the aerated Reactor 17 containing KNO_3 as the nitrogen supplement. Relatively good gas production occurred in terms of the percentage of CO_2 (Reactor 17, page 25). The CO_2 produced was comparable to other nitrogen supplements with a percentage conversion, in terms of mineralization, of 8.4% (Table 2).

All of the remaining experiments conducted aerobically were those receiving chicken litter as the primary source of nitrogen. Reactors 18, 19 and 20 contained a ratio of compost:chicken litter of 1:1, 2:1 and 1:2, respectively (Table 1). The best conversion rate of 7.8 (Table 2), in terms of TVSs, occurred in the reactor with the 1:2 ratio (Reactor 20, Table 1). In all three Reactors, ammonification occurred as determined by a distinct odor of ammonia in the effluent gas and by the increase of pH from 7.4 to 8.1 - 8.8 (Table 2). No visible molds were detected in these reactors. It was established from the cultivation of the molds on both Sabouraud agar and the reactor mix that the visualization of white mycelia growth does not occur when pH values are above 7.5. In Reactor 20 with 1:2 ratio (Table 1), the biological activity occurred over a longer time-period (CO_2 emission profiles) than Reactors 18 and 19. Most of the activity, however, was attributed to the chicken litter and not biological conversion of the pulp and paper waste. Interestingly, the highest temperature of 45°C was reached in Reactor 18 in the first 4 days of operation, dropping to 25-33°C range for the second week and falling to room temperatures thereafter. The temperatures in Reactors 19 and 20 were more consistent with values of 29-38°C for the first 21 days and then cooling-off to room temperature of about 22°C. These were considered reasonably high temperatures since the reactors were uninsulated and had relatively high surface areas. The percentages of CO_2 were clearly the highest in these reactors than any of those previously studied without chicken litter (see figures of reactors 18-20).

The compost to chicken litter mix was again restructured at ratios of 2:1 (reactor 23, a repeat of reactor 19), 1:1 (Reactor 24, a repeat of Reactor 18), 1:2 (Reactor 25, a repeat of Reactor 20) and 3:1 (Reactor 22). Reasonably good correlations were observed for the ratios of 2:1 (Reactors 19 and 23, Table 2) and 1:2 (Reactors 20 and 25, Table 2) with differences in TVS values of 5.6/7.6 and 7.8/9.0, respectively. A larger difference was observed with the duplicates of the ratio of 1:1 in Reactors 18 and 24 with differences in TVS values of 4.7 and 10.7, respectively. The duplicates of each reactor mix were set up some three months apart. One must assume, however, when relatively small samples are used, the variation in the sample may be significant. This problem should disappear on scale-up. Expectedly, the highest CO_2 output occurred with the reactors containing the highest proportion of chicken litter in Reactors 18 & 24 (1:1) and Reactors 20 & 25 (1:2). Nonetheless, the CO_2 profiles are different (see corresponding figures).

Reactor 22 containing a ratio of 3:1 of compost mix and chicken litter produced a broad and continuous output of CO_2 resulting in a mineralization of 12% of the total compost mixture (Table 2). The ratio was repeated twice with very similar results. While the level of CO_2 was less than those with higher concentrations of chicken litter (TVS of 5.6-9.0, Reactors 19, 20, 23, 25 and Table 2) the degree of mineralization was greater in Reactor 22 (Table 2) with its 3:1 ratio.

The highest percentage of mineralization (21.5%) was recorded in Reactor 26 which contained only primary sludge and chicken litter in a ratio of 3:1. This was expected since the bark and grit were excluded with the sawdust of the chicken-litter being sufficient for bulking the compost.

Reactor 21 was established with compost mix to chicken litter in a ratio of 4:1 (Table 1). As expected, the biological activity, measured by CO_2 emissions, was initially high but relatively brief, completing the cycle in 10 days. About 16% of the compost mix was degraded but over a 56 day period as determined by combustion of aliquot samples and quantitating the ash and TVS contents by gravimetric methods (Table 2). The volume of compost was also reduced by 35%. At the end of the 56th day, the compost mix was sprayed with 200 ml of 75 g of NH_4NO_3. The matrix was rebulked with grit and bark (1:1) and added to the compost mix to replace the approximately 35% volume lost. The additional supplement had little effect on the bioconversion rate, increasing the CO_2 emission only slightly (Reactor 21A). At 78 days the CO_2 emission had fallen to near zero. At the 78th day the matrix was removed and sprayed with a 100ml of 60g of KH_2PO_4 (Table 1). A modest increase in CO_2 resulted and appeared to stabilize for the next 20 days (Reactor 21A). At the 100th day, the forced air flow to the reactor was stopped, the reactor flushed with N_2 gas and the reactor sealed. The reactor was then monitored daily for CO_2 and CH_4 production under anaerobic conditions. The CO_2 level remained stable in the closed reactor for an additional 56 days (156th day) with no detectable CH_4 production and only a brief and small gas pressure buildup on three brief occasions. These results support a proposal that addition to the reactants with salt supplements after extended incubation periods are ineffective. This conclusion is further supported by the results presented in the anaerobic digestion study described in the following section.

In none of the reactors was the moisture content of the mixes particularly critical. In all cases where biological activity occurred additional "wetness" occurred. In general, It was found that when the moisture content approached the lower limit of 35% microbial activity was slow and poor reaction rates where obtained. Thus, in all experiments, care was taken to have the starting moisture contents at no less than 40%. When the moisture content at the start of the composting was at 65% and higher, the additional water from the composting caused the mix to be too wet and the porosity diminished. In such cases, air diffusion was limited and in a few trial runs, CH_4 gas was observed in relatively trace quantities in the outflow gas stream. Thus, moisture contents of 45 to 55% were considered as the ideal operating range.

The temperature for optimal composting rates must vary. Most microorganisms with diverse metabolic properties are mesophiles (10- 40°C). These are the major consumers of complex organics. The higher temperatures are also needed for the heat shocking of fungal and bacterial spores and for enhancing activities of thermotolerant organisms that are known to exist. Thus, the microbial activity is one of succession and interspecies activities. No significant thermophilic microbial activity per se was expected because of the nature of the *in situ* cultures adapted to psychrophilic and predominantly mesophilic environments. For optimal activities, the temperature range should naturally be allowed to vary from 20°C to 60°C. Temperatures above 60°C diminish biological conversion rates and if allowed to persist, lengthen the recovery of the microbial activities. The temperatures can be controlled by aeration rates and moisture content. The temperature cycle up to 60°C should last for only 2-3 days if the compost is properly bulked and air diffusion is not limited.

It is felt that much of the bioconverison data (TVS/ash) presented in Table 2 represent values below those that actually existed. The basis for this lies in the difficulty to obtain representative samples on a small scale for measuring ash and TVS. The diverse sizes of wood and bark chips and the fluffy nature of the primary sludge and the high content of sand created technical difficulties. The sand adhered to the particles when wet but easily separated from the particles when dry. Also, ball-milling the particle enhanced the separation of sand from the moderately dense primary sludge and the less dense bark chips. Unfortunately, not all of the sand or consistent amounts of it could be removed by this method. In the first lot of primary sludge obtained from the plant in Olgethorpe, GA, the ash content was between 20 and 30%. For the second lot of primary sludge, the ash content was between 40 and 68%. Significant quantities of sand separated from the samples during the collection, drying and ashing process. When added back to the samples, the conversion rates are significantly higher. In due process, however, such corrections were not made in Table 2.

Anaerobic Compost Reactors.

The mix of pulp and paper waste used in the aerobic composting studies described above was also used in most of the anaerobic experiments. Seven different types of nitrogen compounds and mixes thereof were tested as supplements. All seven sources of nitrogen stimulated microbial activity, but with mixed results. The experimental description of the supplement added to each reactor, the concentrations, and sequences of additions are summarized in Table 1 and are designated as A1-A21. The performances of these reactors are summarized in Tables 3 and 4 (pp31 & 32). The amount of gas pressure produced daily (pp35-46), the total moles of gas produced daily (pp47-65) and the mole percentages of each gas (pp66-80) are given in figure form. All reactors started out under microaerobic conditions but quickly went to anaerobic condition within the first week of operation as detected by oxygen sensitive strips and the loss of O_2 detection by gas chromatography. Nonetheless, the N_2/O_2 detections are not differentiated in the figures. The addition of NH_4NO_3 and NH_4Cl, resulted in predominantly CH_4 and CO_2 production whereas the addition of KNO_3, $NH_4H_2PO_4$,Urea and chicken litter produced principally CO_2. The amount of gas pressure produced are summarized in Table 3 with the daily productions recorded in the corresponding figures. In all reactors containing an addition of NO_3^-, nitrous oxide (N_2O) was produced. The most abundant N_20 was produced in Reactor A11 with only KNO_3 added in the beginning of the incubation period. The results are expressed inthe figures designated A11 (p38,39,56, & 71). For NH_4NO_3 containing mixes in Reactors A1- A5 (Table 1 and corresponding figures), N_2O appeared in relatively trace quantities during the first two weeks of the reaction and disappeared from detection prior to the appearance of CH_4. The detection of N_2 occurred throughout the reaction periods, in all reactors, with considerable variation in relative quantity. Some of the N_2 variation was attributed to contamination from air during sampling, however, the variation was somewhat rhythmical suggesting a combined nitrification-denitrification system in operation like that previously described by Pel et al (Appl. Microbiol. 63:474-481, 1997) under limited oxygen conditions.

The highest total gas pressures were recorded with those reactors containing NH_4NO_3 and NH_4Cl. These reactors also produced gas pressures over the longest time period and with the highest metabolism of substrates (Table 3) and with the largest percentage of CH_4 (Table 4). In Reactors 1-7, 33 to 59% of the starting materials were mineralized during the incubation period (Table 3) as determined by dry weight differences. Interestingly, the odor of the final product of A1 and A2 smelled like fresh barnyard waste whereas the odor of A3 and A4 were more earthy. Perhaps the differences are due to the addition of $NH_4H_2PO_4$ to Reactor A3 and NH_4Cl to Reactor A4 on day 119 of the incubation periods (Table 1, p17). The addition of these salts to Reactor A3 and A4, late in the incubation period, however, had no significant impact on the bioconversion of the existing reactants.

The effects from the addition of NH_4Cl (Reactor A5, Table 1) and $NH_4H_2PO_4$ (Reactor A6, Table 1) and combined NH_4Cl and $NH_4H_2PO_4$ (Reactor A7, Table 1), at a calculated C:N concentration of about 30:1, are summarized in Tables 3 and 4 and the corresponding figures. Gas production in Reactor A6 responded most quickly but dropped off after only 20 days and didn't start generating significant amounts of gas until the 65th day (Fig. A6, p36). Reactor A5 with NH_4Cl started to produce after 35 days and continued for another 35 days but at a relatively low rate. The addition of $NH_4H_2PO_4$ to Reactor A5 (Fig. A5, p36) on the 80th day had no effect on the bioconversion rate. The amount of CH_4 produced was less than 36% of the total gas produced. The dry weight differences between the starting material and the end product, however, showed that a respectable 39.3% of the material was lost during the incubation period. Reactor A6, however, received NH_4Cl on the 80th day resulting in a significant simulation of gas production (Fig. A6, p36). Its percent CH_4 was similar to Reactor A5 and its difference in total weight of the reactants after incubation showed that 44% was lost, only slightly greater than Reactor A5. Reactor A7 with both salts had better gas production (Fig. A7, p36), methane production of 44% and greater utilization of reactants than the reactors containing the individual salts (Tables 3 and 4). The required incubation times for the reactors was the shortest for Reactor A7 with only 90 days as compared to 120 days for Reactors A3, A4, A5 and A6 and 150 days for Reactors A1 and A2. The results suggest that mixed salts added initially provide better enrichment conditions. However, the results from Reactor A8 which contained all of the salts plus urea in a C:N ratio of 30:1gave poorest results. High gas production started to occur on the 7th day in Reactor A8, peaking at the 17th day but soon dropping to no pressure and then neg. pressure on the 22nd day. The pH of the reactants was 6.06 on day 23. Thereafter, the gas pressure oscillated in five day cycles (Fig A8, p69) with gas compositions of principally CO_2 with only detectable amounts of H_2, N_2O, and CH_4. The bioconversion reduction of the reactants was only 15%. The final product smelled strongly of butyrates indicating that one of the predominant microbial enrichments was possibly the fermentative Clostridia. The same mixture with the nitrogen sources was also placed in a 20 L reactor in a solid form and sealed. This reactor labeled A9 reflected what one may find in a landfill if diverse nitrogen salts were added. Essentially, nothing had happened after 85 days of incubation. CO_2 was the major head-space gas but with no gas pressure or detectable changes occurring. These results are comparable to a similar set up of reactants taken from aerobic compost Reactor 21 (Table 1), spiked with nitrogen supplements and sealed after flushing with nitrogen gas to remove air from the chamber. This Reactor produced no indication of further metabolic activity.

Reactor A10 was supplemented with only urea. No gas pressure changes were detected until the 7th day when a negative pressure was recorded (fig. A10, p37). The reactor was unsealed and pH recorded at 8.9. With a strong smell of ammonia coming from the flask, it was assumed that ammonification occurred with the NH_4^+ dissolving in the aqueous mixture and creating the negative pressure. The pH was adjusted to 7.64 with H_2SO_4 and the reactor resealed. Thereafter, a positive gas pressure was recorded for the next 25 days. No CH_4 was detected (Table 3), however, a respectable reduction (28%) of reactants was recorded (Table 3). The odor of the final product was once again that of specific fermentative bacteria with a strong smell of butyrates.

Reactors A-11 supplemented with KNO_3 and A12 reactants mixed with Chicken litter are described in the corresponding figures on pp39,40,56,57,71 and 72. The only gases detected in A12 were N_2 and CO_2 with CO_2 comprising 80% of the total gas emitted for the first 50 days and then a moderate amount of CH_4 produced thereafter. Reactor A11 differs in that a significant fraction of the emitted gases was N_2O for an extended period. After the NH_4Cl spike, the CH_4 has gradually increased with the inverse production of CO_2. The final results are reported in Table 3.

Reactors A13 and A14 were established with the standard mixture of pulp, fiber, bark, grit and ash with no exogenous nitrogen added. These represented controls. The two preparations differ, however, in that the primary sludge used in bioreactor A13 was taken from a barrel containing high moisture content (loaded in the barrel 12 months previously) and demonstrated fermentation processes. The primary sludge used in bioreactor A14 was taken from a more recently loaded Barrel that had little moisture and no evidence of biological activities (see Table 1B). Bioreactor A14 showed no biological activity for the first 35 days and it was subsequently terminated. Bioreactor A13, however, showed biological activity within the first 2 days of incubation. The amount of biological activity in A13 was relatively good in comparison to other Reactors supplemented with exogenous nitrogen. Most of the gases produced was CO_2 with low amounts of CH_4 occurring at 45 days. At the end of 45 days the majority of the contents of Reactor A13 were used as the starter cultures for reactors (A19-A21). The remnants of A13 as well as several others established just like it are being held for seed cultures for future reactors.

Reactors A16 and A18 were structured like A13 with primary sludge taken from the barrel with biological activity but different from A13 in that these two reactors received NH_4Cl in A18 and $NH_4H_2PO_4$ in A16. For comparison to the effects from starting with an active biological culture vs. one not biologically active, Reactor 17 was established with dried primary (nonactive) sludge but with NH_4Cl supplement like A18. A17 was terminated after 30 days as it was just starting to ferment (results not included in this report). This experiments was performed only to establish the differences in lag time of seeded and unseeded cultures. A18 and A16 started to produce gas within hours of the start of its incubation period. A18 differed from A16 in that CH_4 was produced almost immediately whereas no CH_4 occurred in A16 until much later and even then at relatively low quantities. A16 continues to produce high gas levels at day 45 but still with relatively low CH_4 yields (Fig. 16A, pp42, 61 and 76). A18's level of CH_4 reached 50% of the total gas within 20 days of incubation, however, the gas pressures began to drop shortly thereafter. A small supplement of NH_4Cl stimulated methanogenesis. The periodicity of the supplements are given in Table 1, with results described in Figures on pp 43, 62 & 76. The summary of the results of expeiment A18 is given in Tables 2 & 3 (pp33 & 34).

Three addition reactors have been recently established with nonbiologically active mix (A19, A20, A21) with different NH_4Cl concentration and seeded with active cultures from bioreactor A13. The nitrogen supplement was determined to be at a C:N ratio 45:1, 60:1 and 75:1 for Reactors A19, A20 and A21, respectively. The biological activity was almost immediate with highest gas pressures produced first in Reactor A21 (Fig. A21, p44), the reactor with the least amount of NH_4Cl. The original intent was to supplement the reactors with additional salts through the course of their incubation periods. The results, however, showed excellent methanogenic activity for A21 with activity equal to or better than Reactors A20 and A19 with higher nitrogen supplements (Fig. A20 & A21, pp63-64 and pp78-80). The summary of the results are given in Tables 3 & 4. At 80 days, A21 has just about stabilized while A20 and A19 have moderate gas production, attributed to the additional supply of nitrogen. A19, however, is currently producing principally CO_2. The dry weight conversions values of these three reactors has not been completed. Further experiments will have to be established to determine the concentration of nitrogen needed to stimulate methanogenesis and the subsequent feeding strategy

Nitrogen, carbon and hydrogen analyses were performed for 7 of the anaerobic reactors. The results are summarized in Table 5. The results are mixed. The results are suspect since only a few milligrams were analyzed from each preparation and questions remain about the representation of the samples analyzed. The data support the data presented in Table 3 that most of the nitrogen stayed in the reactor. The CHN analyses (Table 5) support the end-product results reported in Table 3 in terms of the high ash content and the percent converted in the reaction.

The market analyses of the value of CH_4 produced was calculated for each of the reactors operated anaerobically (Table 4). The BTU values of the higher producers of CH_4 were also calculated on a dry weight basis and organic content basis. The highest percentages of methane gas (>55%) was observed in Reactors A1-A4 and A21 (Table 3) which contained NH_4NO_3 (A1-A4) or NH_4Cl (A21) at the start (t_o) of the incubation period (Table 4), but in total liters of CH_4, the best producers were A4 and A18-21. Additions of NH_4NO_3 later in the incubation periods were ineffective. Reactors A1-A5 were essentially replicates with the exception that increasingly more water/unit weight of reactants was added with A1 receiving the least proportionate amount of water (Table 1). The data clearly shows a relationship between the greatest amount of gas being produced and the more liquid properties of A3 and A4. Better total reduction in reactants, however, occurred in Reactors A1 and A2. Obviously there is a difference in biomass and fermentation product accumulation relative to the dynamics of the system some of which are volatized during the ashing process. An important piece of information learn at this point in the study is the stimulation of the methanogens at higher C:N ratios, concentrations significantly removed from the reported ideal ratio of 30:1.

As for the nitrogen sources, the general conclusions are:

Chicken Litter stimulates organisms that produce CO_2,
Urea stimulates organisms that produce CO_2,
KNO_3 stimulates organisms that produce CO_2 and N_2O (in decreasing order)
NH_4Cl stimulates organisms that produce CH_4 and CO_2 (in decreasing order)
$NH_4H_2PO_4$ stimulates organisms that produce CO_2 and low levels of CH_4
NH_4NO_3 stimulates organisms that produce CH_4, CO_2 and N_2O (in decreasing order)
NH_4SO_4 stimulates organisms that reduce sulfur

All of the nitrogen sources used to supplement the reactions resulted in significant mineralization and reduction in volume of the pulp and paper mill waste. The lowest bioconversions occurred with the additions of Urea, chicken litter and KNO_3.

Measurement of Microbial Populations.

A general enumeration of the microbial populations was conducted with the reactants of Reactors 8, 10, 11, and 12 containing different quantities of NH_4NO_3. The cultivation media used was chosen on the basis of testing the relative fastidiousness of bacteria on 1/10 and 1/50 Trypticase Soy Agar (Table 6) and for fungi on potato dextrose agar containing the antibiotic streptomycin positive bacteria (Table 6). Samples were withdrawn from the reactors for cultivations at the start of the incubation period (t_o), at day 12 (t_{12})of the incubation and when the incubation was stopped at 31 days (t_{31}). At t_o the number of colony forming units (CFU) of microorganisms was relatively low in comparison to the number of colonies at t_{12} and t_{31}. As expected the highest CFU was at t_{12} since numerous protozoa and insects appeared in the reactors after t_{12} samples were taken and which normally feed on the microbial populations.

Media was also prepared with agar and cellulose or biocellulose as the only carbon source to test the ability of the population to produce cellulase. Two different molds were isolated that had the ability to grow on cellulose while numerous CFUs were obtained from the biocellulose medium. Mycelia cultures were also observed in the anaerobic reactors growing on "chips" of wood and bark attached to the sides of the cultivation flasks. No identities of the organisms were attempted at this time.

Description of Sample Preparation **Table 1A**

REACTOR	INGREDIENTS		ADDITIVES
1	Primary Sludge	3	pH adjusted to 7.2-7.5 on day 10 with 200 ml H_2SO_4 and 100 ml HNO_3
	Fly Ash	1	
	old Grit	2	
	Old Bark	1	
2	Primary Sludge	3	1% Urea added on day 6
	Old Grit	1	
	Old Bark	1	
3	Primary Sludge .	3	Fly Ash was adjusted to pH 7.2 with 1 M H_2SO_4
	Old Grit	2	
	Fly Ash	0.01	
	Old Bark	1	
4	Same as 3		50 g of NH_4NO_3 to 5.46 Kg of mix, added as spray in 100 ml H_2O
5	Same as 3		100 g of NH_4NO_3 to 5.5 kg of mix, added as spray in 200 ml H_20
6	Same as 3		50 g of NH_4NO_3 plus the addition of 200 ml of undiluted Ecosystem Plus^R
7	Same as 3		50 g of NH_4NO_3 plus 20 ml of Formula #615 Composting^R in 100 ml of water sprayed on 5.56 Kg of mix.
8	Same as 3		30 g of NH_4NO_3 calculated to give a C:N ratio of 123:1
9	Same as 3		50 g of NH_4NO_3 in 100 ml H_2O to 11.5 kg to give a C:N ratio of 77:1
10	Same as 3		60 g of NH_4NO_3 to 11.5 Kg to give a C:N ratio of 64:1

11	Same as 3	90 g of NH_4NO_3 to 11.5 Kg give a C:N ratio of 43:1
12	Same as 3	120 g of NH_4NO_3 to 11.5 Kg to give a C:N Ratio of 32:1
13	Same as 3	72.73 g of NH_4Cl in 200 ml H_2O to 5.5 kg of mix
14	Same as 3	100 g of $NH_4H_2PO_4$ in 400 ml H_2O on 5.5 kg of mix
15	Same as 3	170 gm NH_4Cl + 30 gm $NH_3H_2PO_4$ + 100 gm NH_4NO_3 + 50 gm Urea in 600ml H_2O sprayed on 18.2 kg of mix
16	Same as 3	36 gm NH_4NO_3 was added to 4L H_2O and used to suspend 1.9 kg of mix at 40% moisture
17	Same as 3	400 gm KNO_3 to 1.9 kg of mix
18	Same as 3	Mixed with chicken litter at a ratio of 1:1
19	Same as 3	mixed with chicken litter at a ratio of 2:1
20	Same as 3	mix with chicken litter at a ratio of 1:2
21	Same as 3	mix to Chicken litter ratio 4:1. After 56 days, mix was bulked with old bark and grit (1:1) at 3:1 ratio and sprayed with 75 gm NH_4NO_3. After 78 days, sprayed with 60 gm KH_2PO_4. (see 21A)
21A	Composted ingredients of mixture 21	After 100 days, the reactor was flushed with N_2 and sealed for anaerobic growth.

22	Same as 3	mix to chicken litter ratio 3:1
23	Same as 3	mix to chicken litter ratio 2:1
24	Same as 3	mix to chicken litter ratio 1:1
25	Same as 3	mix to chicken litter ratio 1:2
26	Primary sludge	mix to chicken litter ratio of 3:1

Table 1B

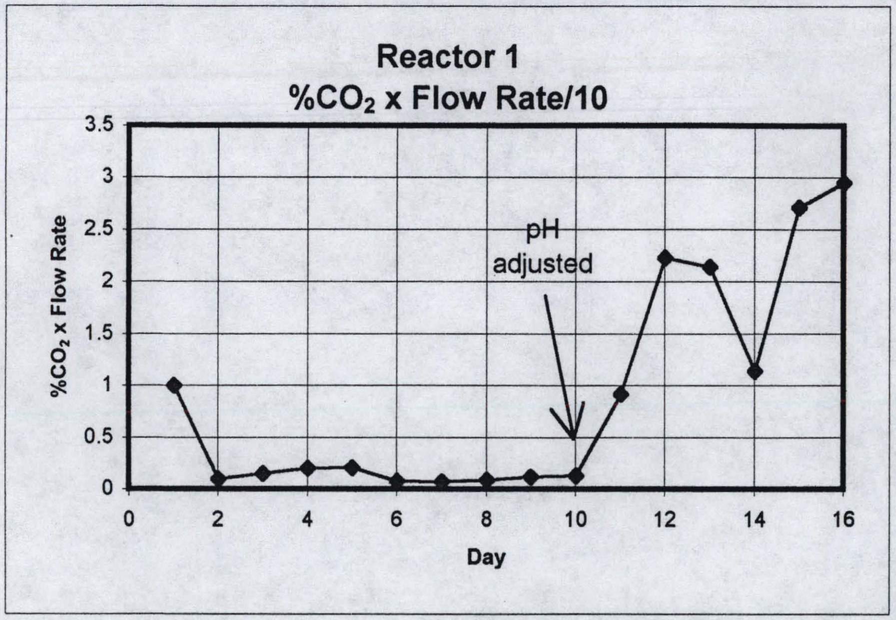
SAMPLE	INGREDIENTS	ADDITIVES
A1	Same as 3	1,874.95 g of mix with 30.28 g NH_4NO_3 in 2 L H_2O.
A2	Same as 3	953.11 g mix plus 23.17 g NH_4NO_3 in 2 L H_2O. 20 ml of Spizizen mineral medium was added at day 141.
A3	Same as 3	630.06 g of mix with 17.36 g NH_4NO_3 in 2 L H_2O. 5 gm $NH_4H_2PO_4$ in 20 ml H_2O was added at day 119.
A4	Same as 3	625.49 g of mix with 16.43 g NH_4NO_3 in 2 L H_2O. 5 gm NH_4Cl in 20 ml H_2O was added at day 119.
A5	Aliquot of sample 13	612 g aliquot of #13 with an additional 0.5% NH_4Cl in 1 L H_2O. 5 gm $NH_4H_2PO_4$ in 20 ml H_2O was added on day 81.
A6	Aliquot of sample 14	415.23 g aliquot of #14 with an additional 0.5% $NH_4H_2PO_4$ in 1 L H_2O. 5 gm NH_4Cl in 20 ml H_2O added on day 81.
A7	mixed aliquots of samples 13 & 14	204.5 g #14 + 211.75 g # 13 with an additional 0.25 % each of NH_4Cl & $NH_4H_2PO_4$ in 2 L H_2O. 0.5 gm NH_4NO_3 in 20 ml H_2O added on day 81.
A8	Aliquot of sample 15	663 gm suspended in 1.5 L H_2O
A9	Aliquot of sample 15	6.4 kg loaded and sealed with no H_2O addition

A10	Same as 3	647 gm + 10 gm urea in 1.5L H_20
A11	Same as 3	636.l gm + 25.2 gm KNO_3 in 1.8L H_20 + 5 gm NH_4Cl on day 46 and 2 gm on day 85
A12	Same as 3	488 gm + 184 gm Chicken Litter in 1.5 L H_2O
A13	Same as 3	698.4 gm (no nitrogen added), wet and biologically active
A14	Same as 3	654 gm of dried mix with no nitrogen added
A15	Same as 3	558.8 gm + 14.6 gm $(NH4)_2S0_4$ in 1.5 L H_20
A16	Primary sludge only	1940.1 gm of wet active sludge + 50 gm $NH_4H_2PO_4$ in 1.3 L H_20
A17	Primary sludge only	1104.1 gm dried sludge + 20 gm NH_4Cl in 1.7 L H_20
A18	Primary sludge only	2179.5 gm active sludge + 25 gm NH_4Cl + 1 L H20 +2 gm NH4Cl on day 35
A19	Same as 3	938.4 gm + 500 mL from reactor 13 + 12 gm NH_4Cl + 1.8 L H_20
A20	Same as 3	927.4 gm + 500 mL from reactor 13 + 9 gm NH_4Cl + 1.8 L H_20
A21	Same as 3	945.7gm + 500 mL from reactor 13 + 7.2 gm NH_4Cl + 1.8 L H_20

Data Summary of Aerated Compost* Table 2

Sample	Moisture % t_0	Moisture % t_f	pH t_0	pH t_f	Ash t_0	Ash t_f	TVS t_0	TVS t_f	Converted %
1	44.0	47.0	8.9	7.3	30.1	31.8	69.9	68.2	1.7
2	47.0	50.1	7.4	8.6	28.8	32.4	71.2	67.6	3.6
3	45.0	46.0	7.2	7.4	28.1	30.1	71.9	69.8	3.8
4	43.9	63.2	7.7	7.5	23.1	26.6	76.8	73.4	3.4
5	41.5	64.2	7.4	7.4	20.0	24.4	80.0	75.6	4.4
6	58.1	56.8	7.9	7.5	21.4	25.2	78.6	74.8	3.8
7	58.5	53.9	7.6	7.0	21.1	23.7	78.9	76.1	2.8
8	63.3	65.5	7.0	7.6	27.3	29.9	72.7	70.1	2.6
9	40.3	46.3	7.5	7.6	22.7	28.7	77.3	71.3	6.0
10	63.3	65.9	7.0	7.4	27.3	31.6	72.7	68.4	4.3
11	63.3	66.1	7.0	7.5	27.3	35.2	72.7	64.8	7.9
12	63.3	64.0	7.0	7.3	27.3	32.4	72.7	67.6	5.1
13	59.6	61.0	7.4	6.8	37.8	43.6	62.2	56.4	5.8
14	46.9	61.8	6.8	7.2	39.2	47.4	60.8	52.6	8.2
15	49.1	67.5	7.0	7.2	45.6	55.8	54.4	44.2	10.2
16	NA	NA	7.5	NA	45.6	NA	54.4	NA	NA
17	52.0	53.8	7.4	7.1	45.6	54.0	54.4	46.0	8.4
18	35.0	38.9	7.5	8.3	26.6	31.3	73.4	68.7	4.7
19	53.1	37.7	7.8	8.1	26.6	29.2	73.4	70.8	5.6
20	41.9	49.0	7.6	8.8	19.4	27.2	80.6	72.8	7.8
21	45.6	64.4	7.8	7.3	24.9	40.9	75.1	59.1	16.0
22	52.0	51.6	7.6	9.1	21.7	33.7	78.3	66.3	12.0
23	53.8	65.0	7.9	8.9	21.6	29.2	78.4	70.8	7.6
24	50.3	60.4	8.0	8.9	21.0	31.7	79.0	68.3	10.7
25	42.5	67.8	8.2	8.9	19.4	28.4	80.6	71.6	9.0
26	60.5	72.9	7.8	6.7	19.4	40.9	80.6	59.1	21.5

*Sample numbers correspond to Reactor numbers identified in Table 1
t_0 and t_f represent time zero and final time, respectively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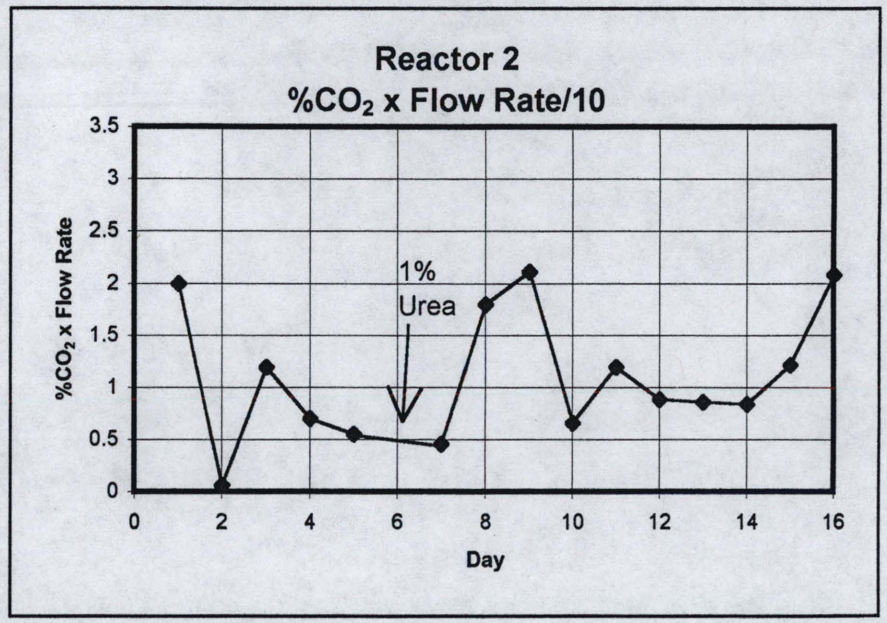
%CO_2 x Flow Rate
Day
pH
adjusted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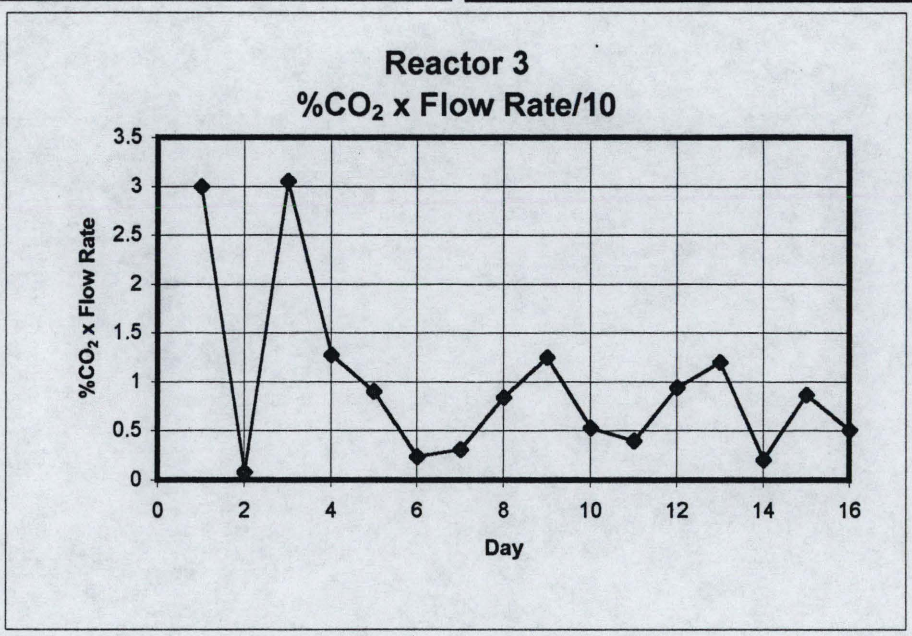
Day
1%
Urea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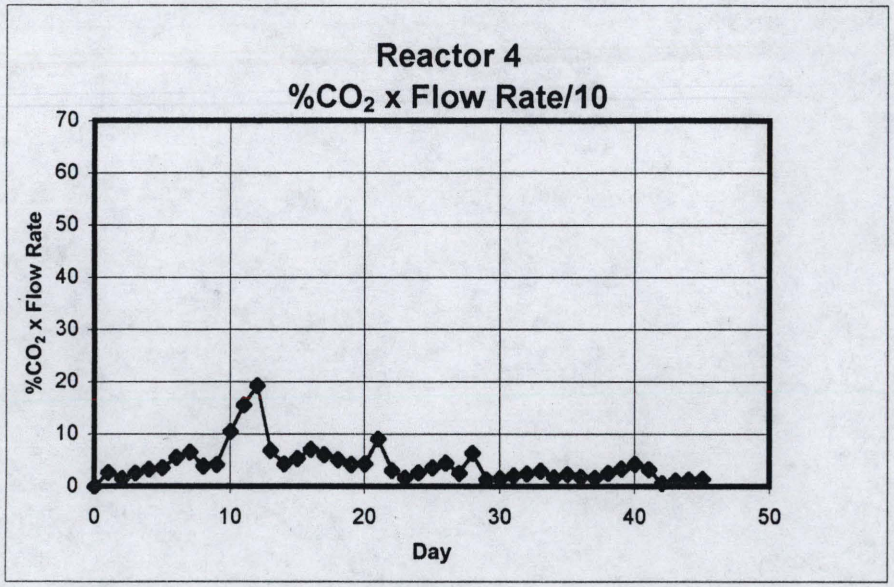
Day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Day
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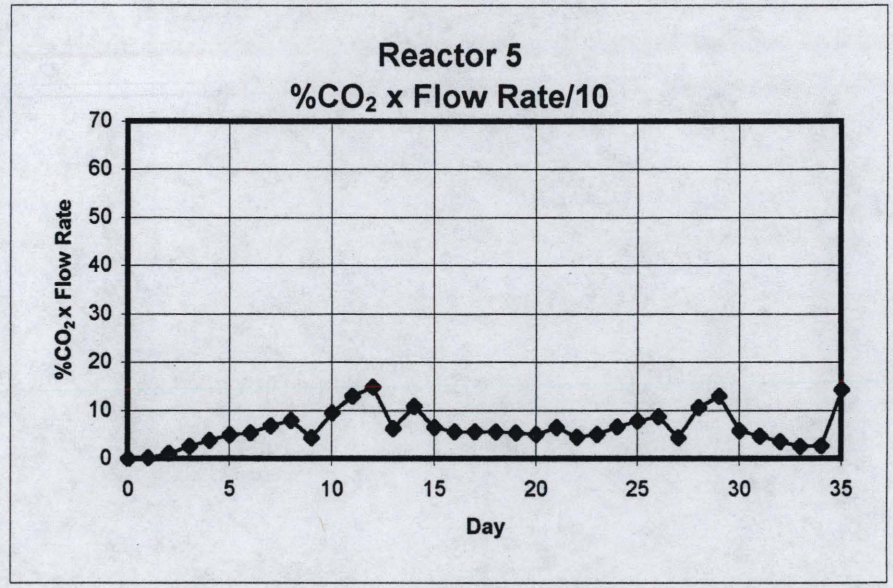
10
20
30
40
50
60
70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Day
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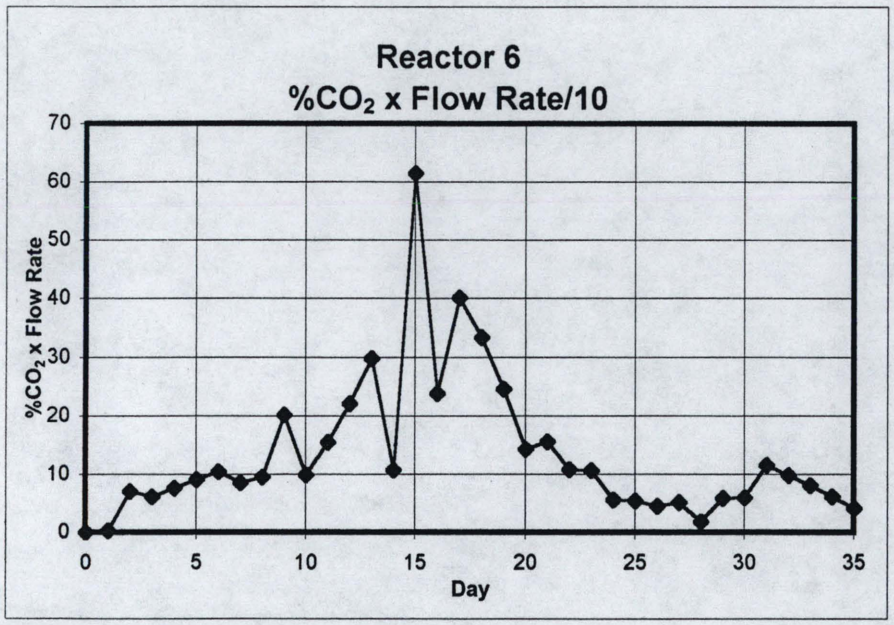
5
10
15
20
25
30
35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Day
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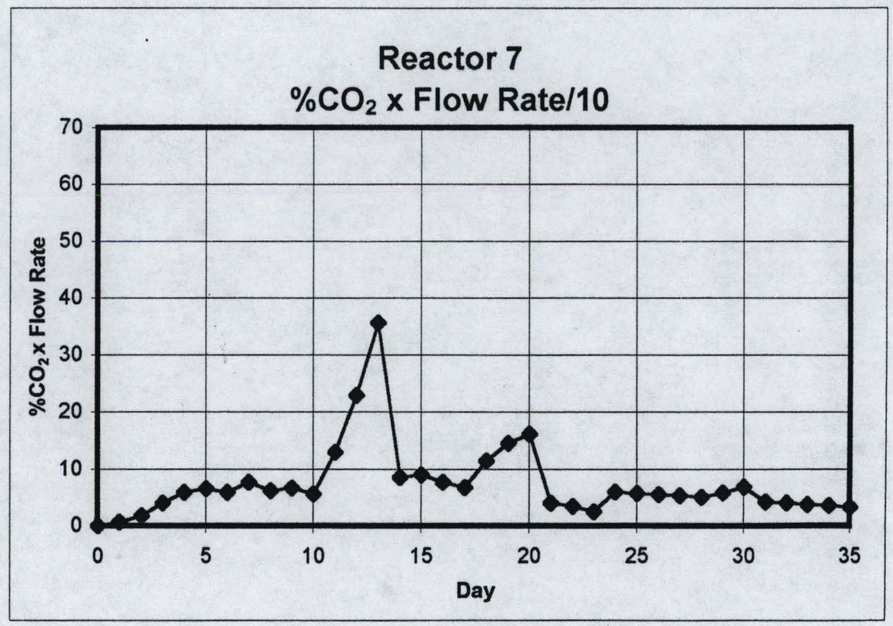
5
10
15
20
25
30
35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Day
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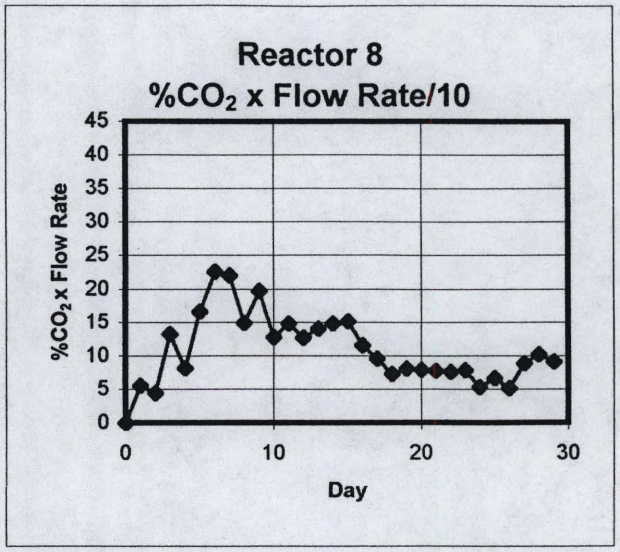
5
10
15
20
25
30
35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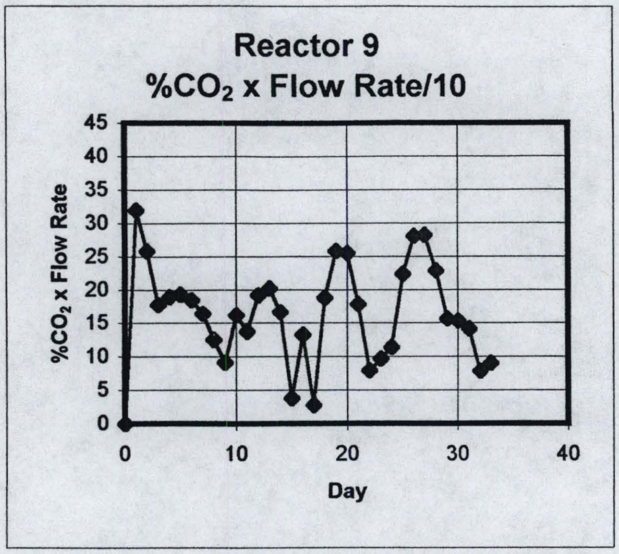
%CO2 x Flow Rate
Day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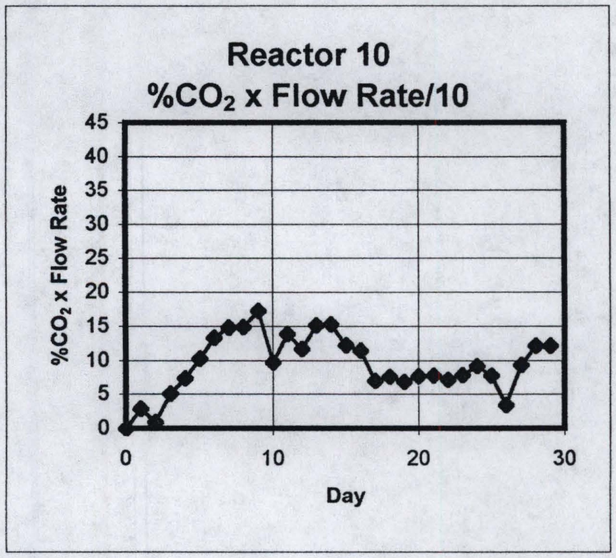
Day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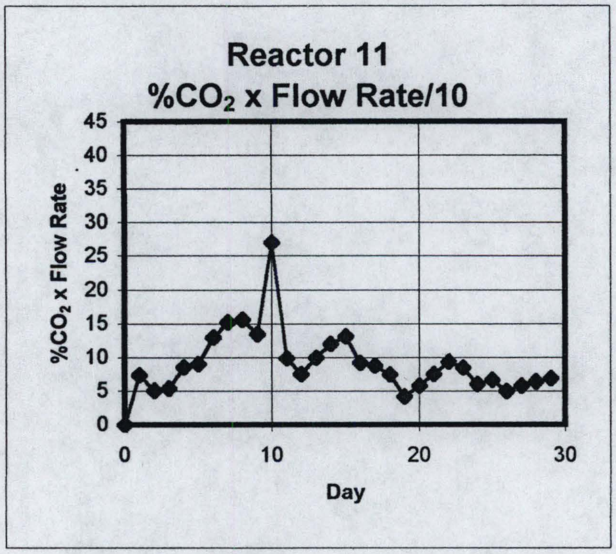
Day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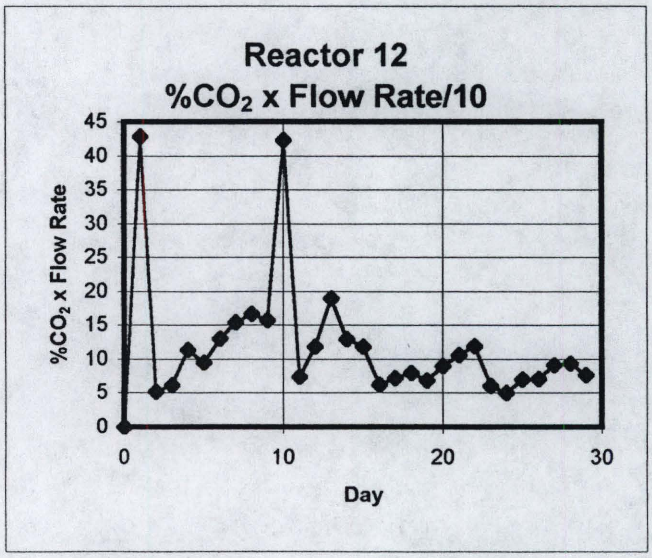
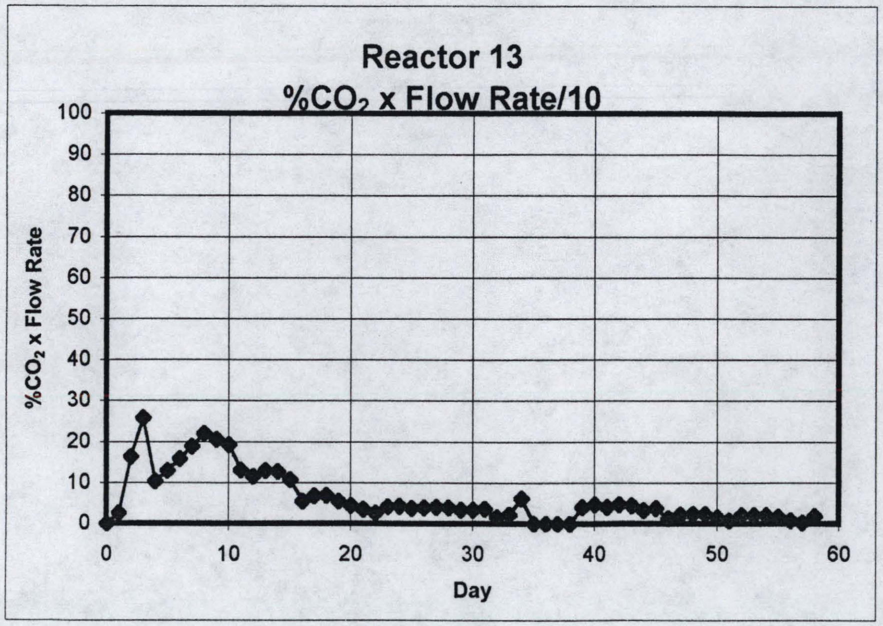
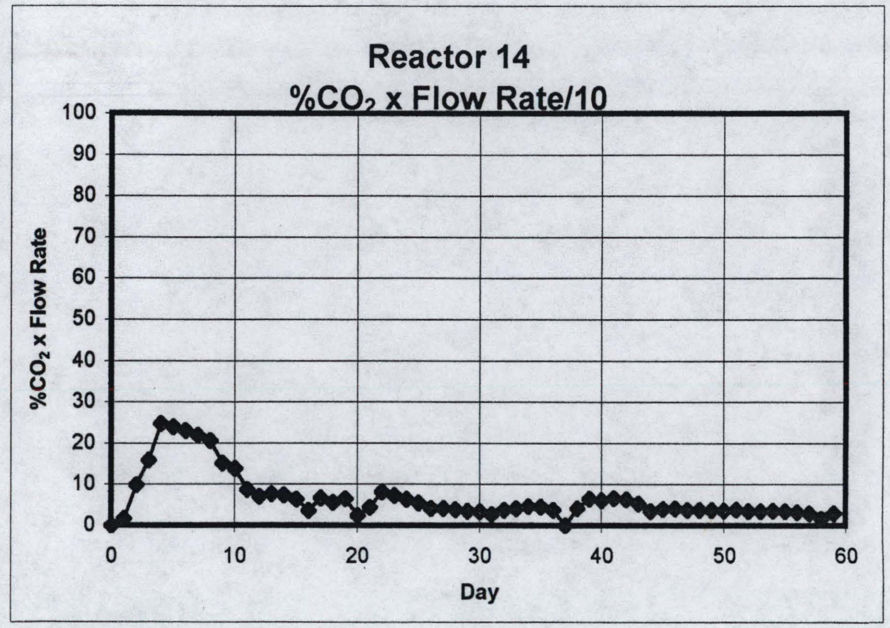
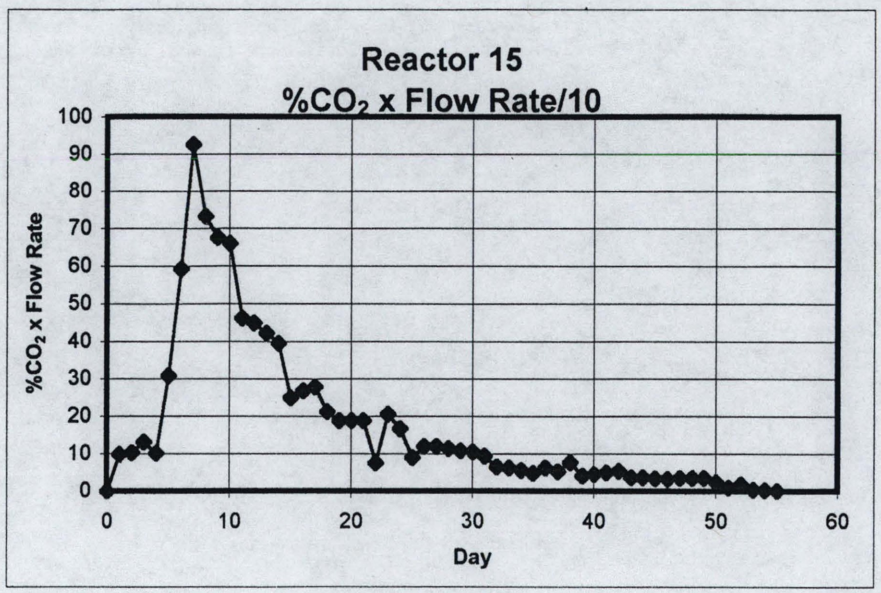
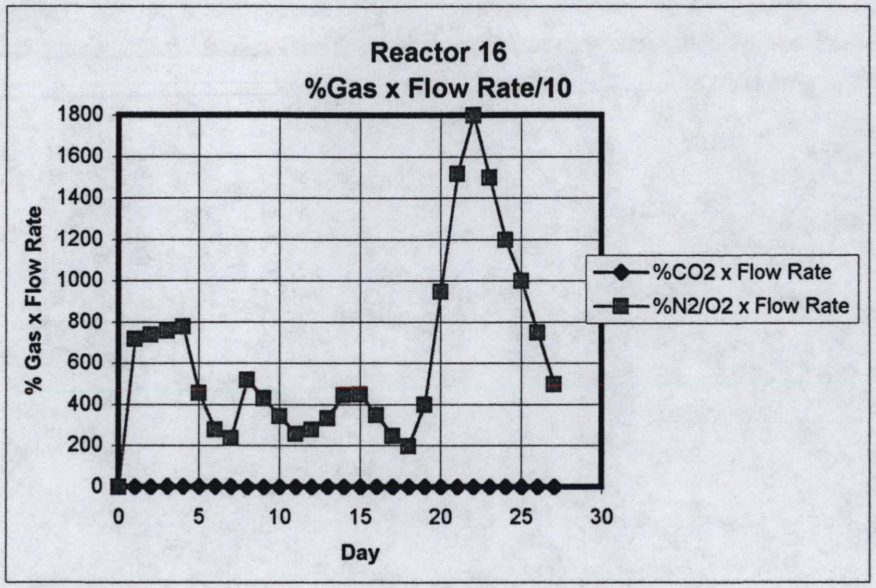
Day


Reactor 12
%CO2 x Flow Rate/10
%CO2 x Flow Rate
Day

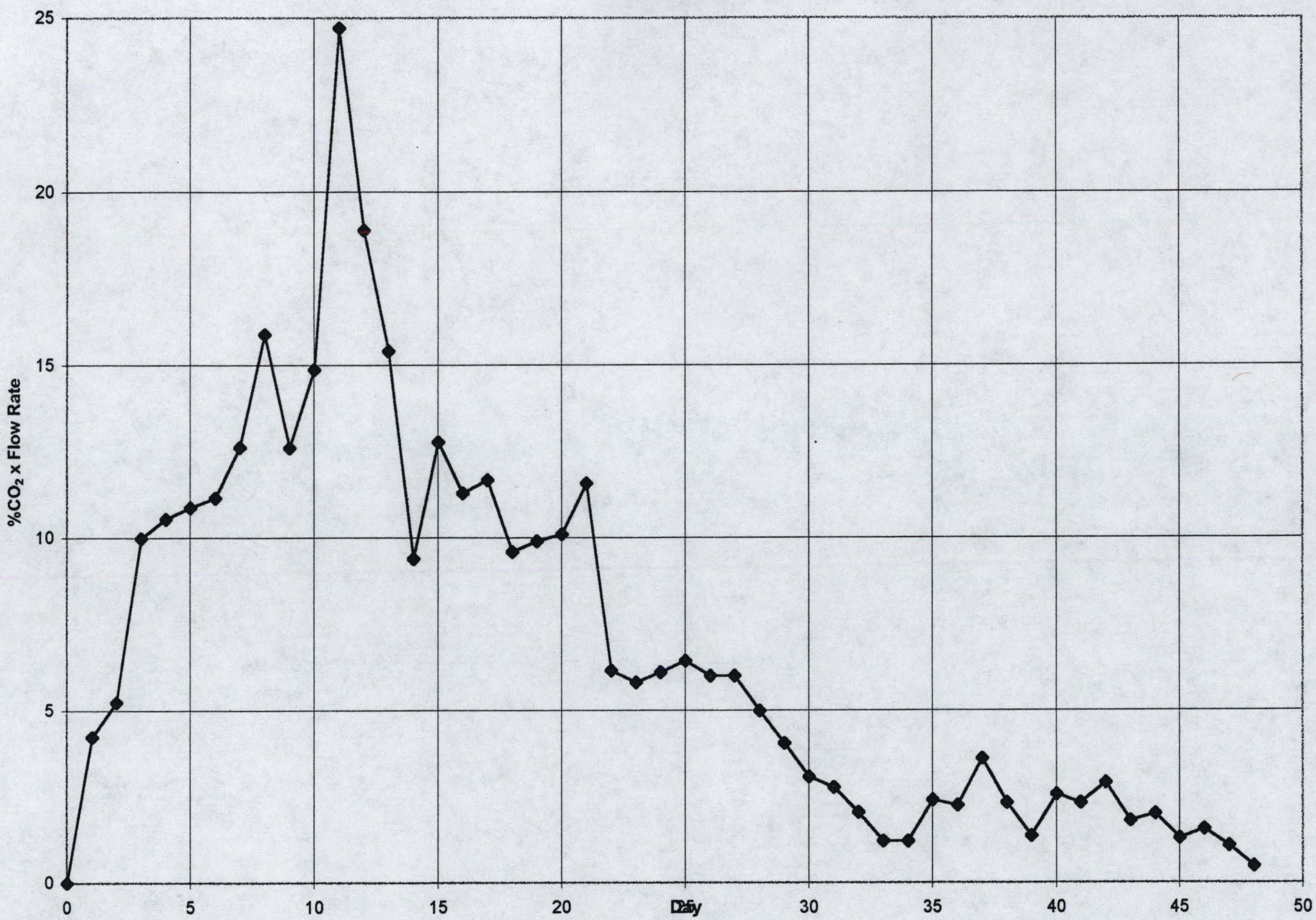

Reactor 13
%CO2 x Flow Rate/10
%CO2 x Flow Rate
Day
0
10
20
30
40
50
60
70
80
90
100

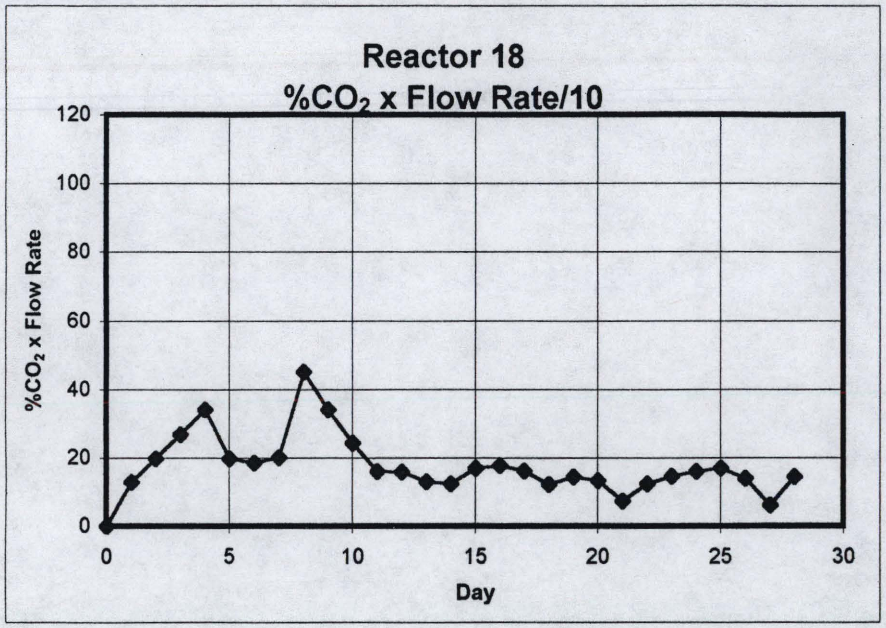

Reactor 14
%CO2 x Flow Rate/10
%CO2 x Flow Rate
Day
0
10
20
30
40
50
60
70
80
90
100

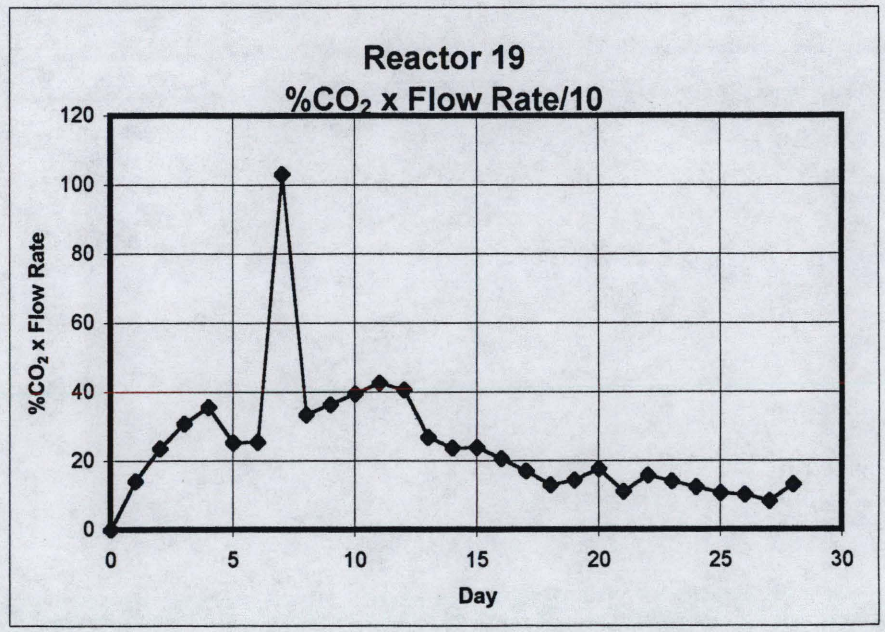

Reactor 15
%CO2 x Flow Rate/10
%CO2 x Flow Rate
Day
0
10
20
30
40
50
60
70
80
90
100

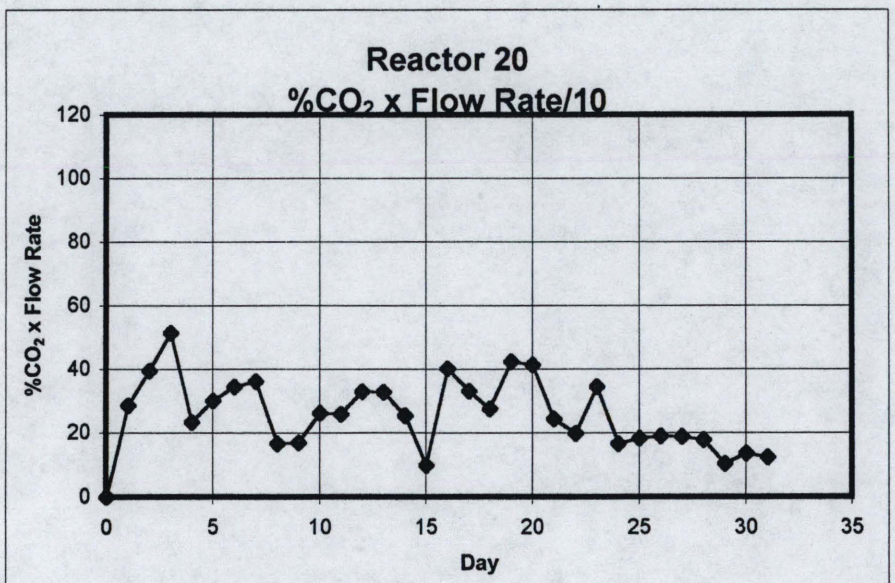

Reactor 16
%Gas x Flow Rate/10
% Gas x Flow Rate
Day
%CO2 x Flow Rate
%N2/O2 x Flow Rate
0
200
400
600
800
1000
1200
1400
1600
1800
0
5
10
15
20
25
30

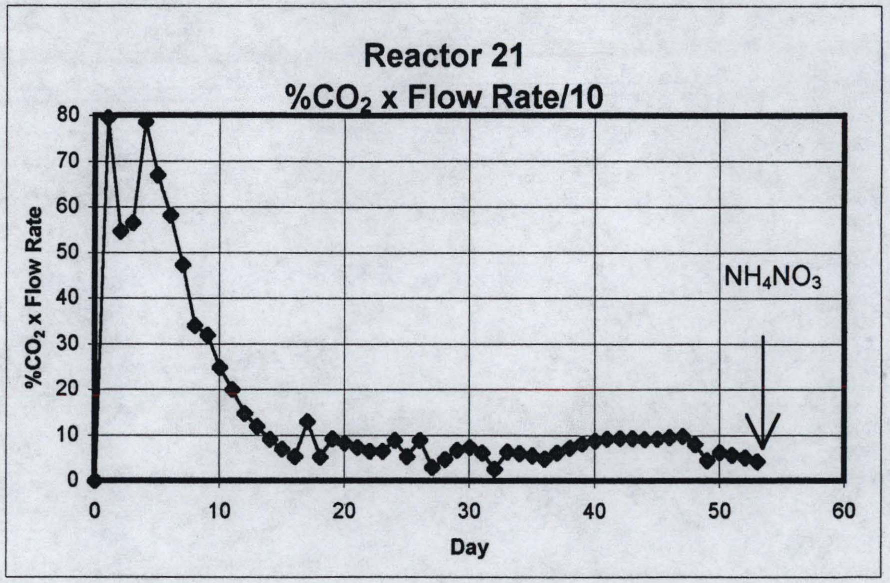
Reactor 17
$\%CO_2$ x Flow Rate/10

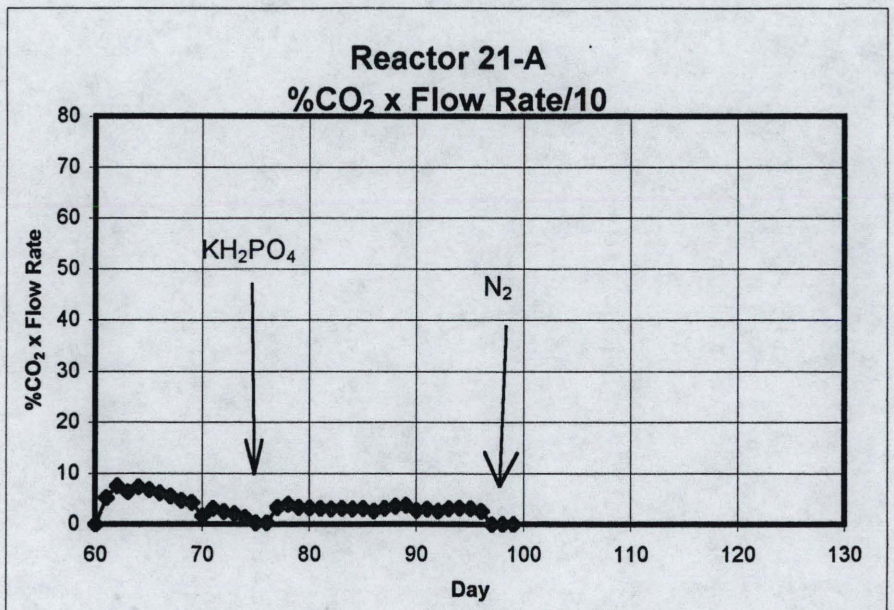


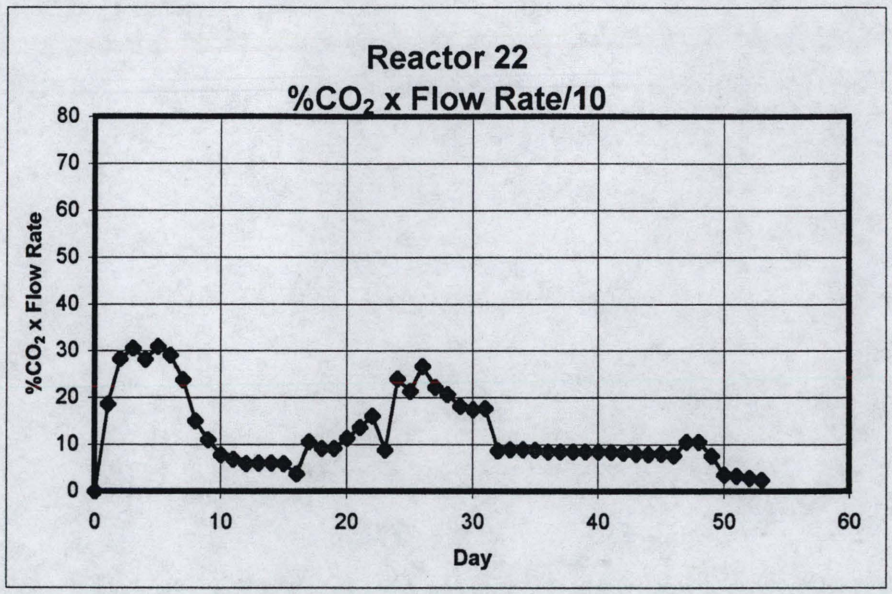

Reactor 18
%CO2 x Flow Rate/10
%CO2 x Flow Rate
Day
0
20
40
60
80
100
120
0
5
10
15
20
25
30

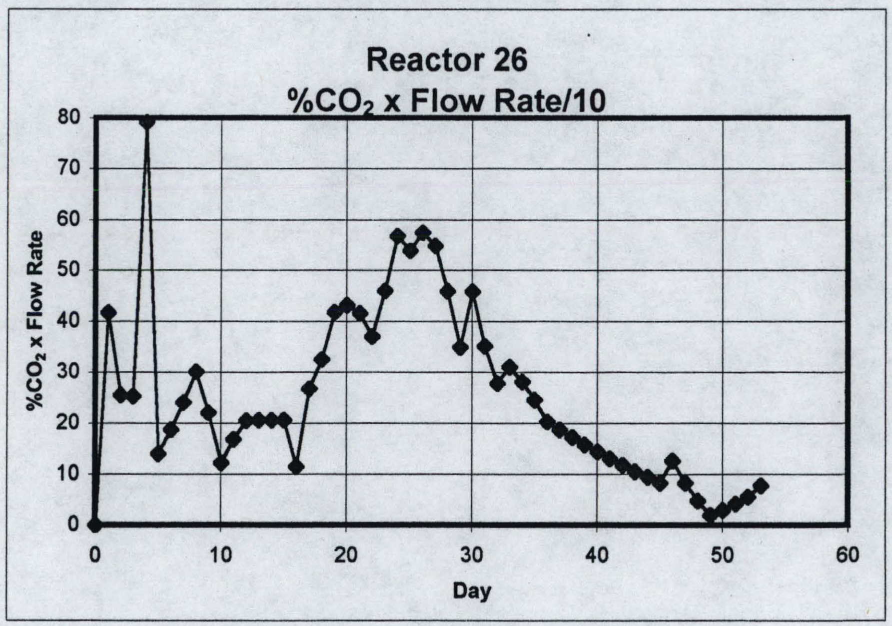

Reactor 19
%CO2 x Flow Rate/10
%CO2 x Flow Rate
Day
0
20
40
60
80
100
120
0
5
10
15
20
25
30

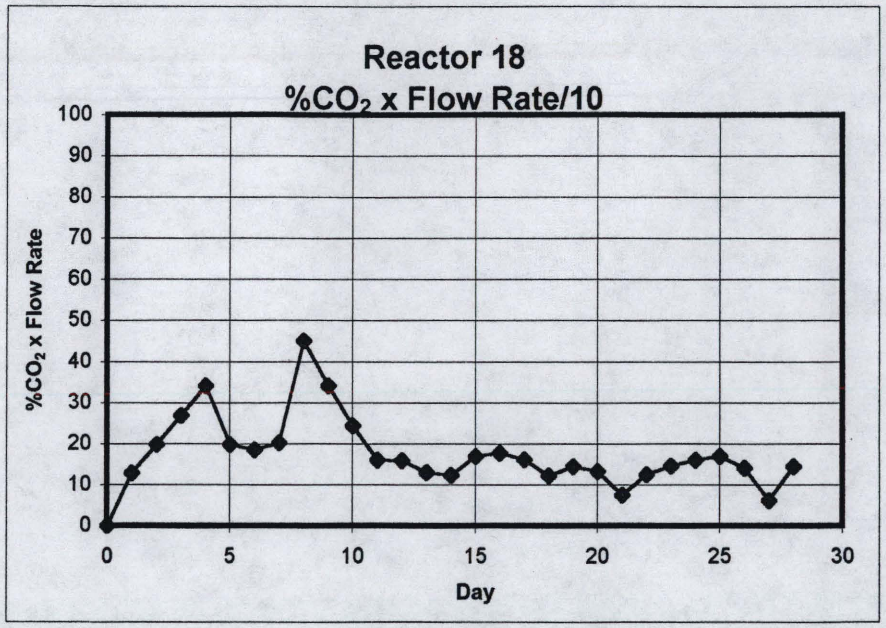

Reactor 20
%CO2 x Flow Rate/10
%CO2 x Flow Rate
Day
0
20
40
60
80
100
120
0
5
10
15
20
25
30
35

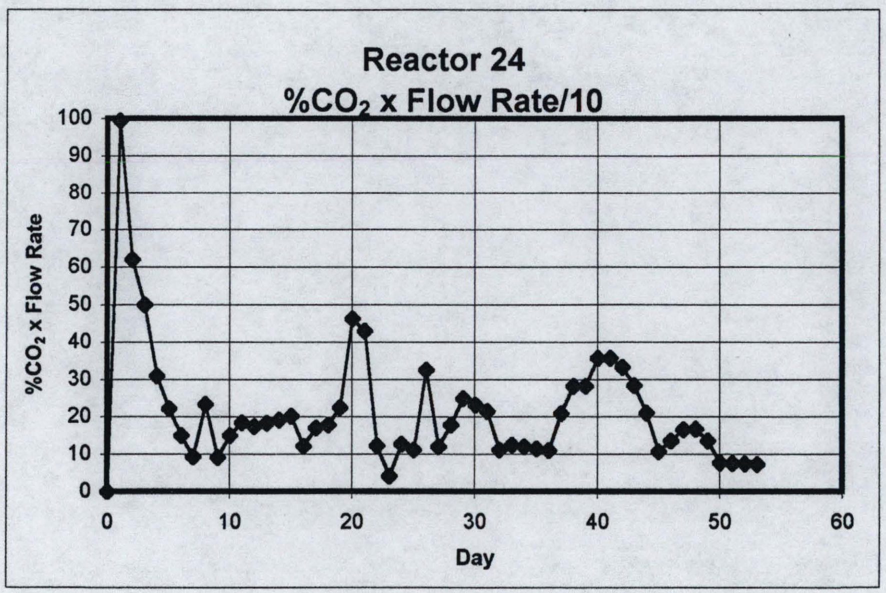

Reactor 21
%CO_2 x Flow Rate/10
%CO_2 x Flow Rate
Day
NH_4NO_3
0
10
20
30
40
50
60
70
80

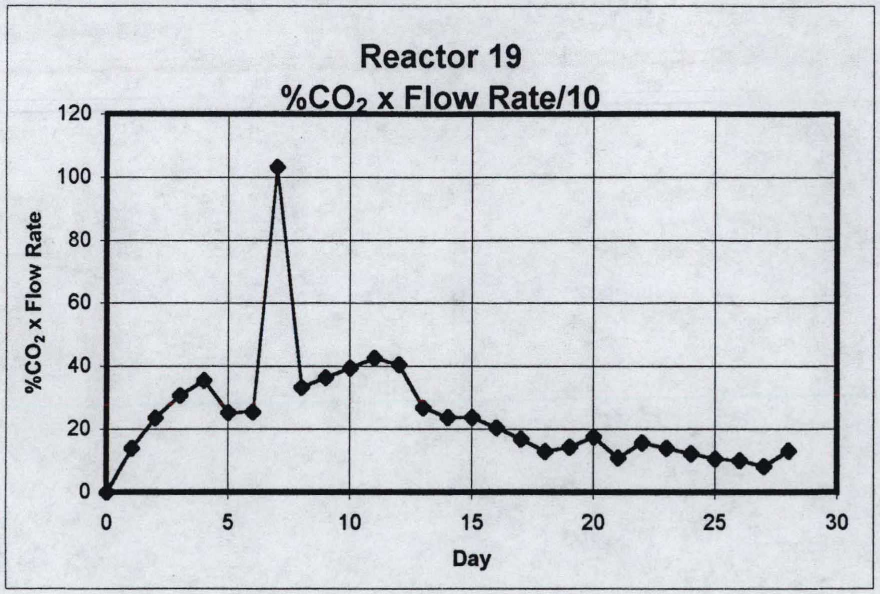

Reactor 21-A
%CO_2 x Flow Rate/10
%CO_2 x Flow Rate
Day
KH_2PO_4
N_2
60
70
80
90
100
110
120
130

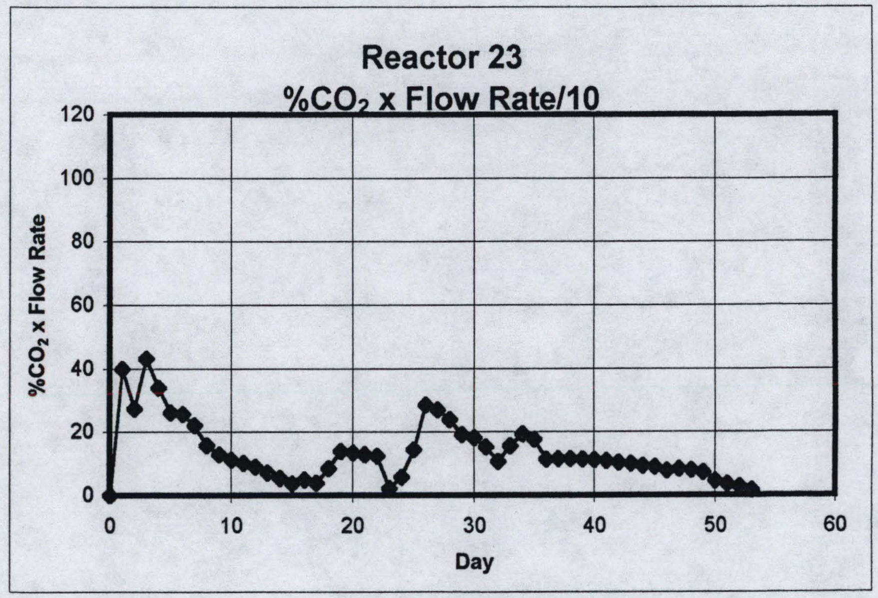

Reactor 22
%CO_2 x Flow Rate/10
%CO_2 x Flow Rate
Day
0
10
20
30
40
50
60
70
80

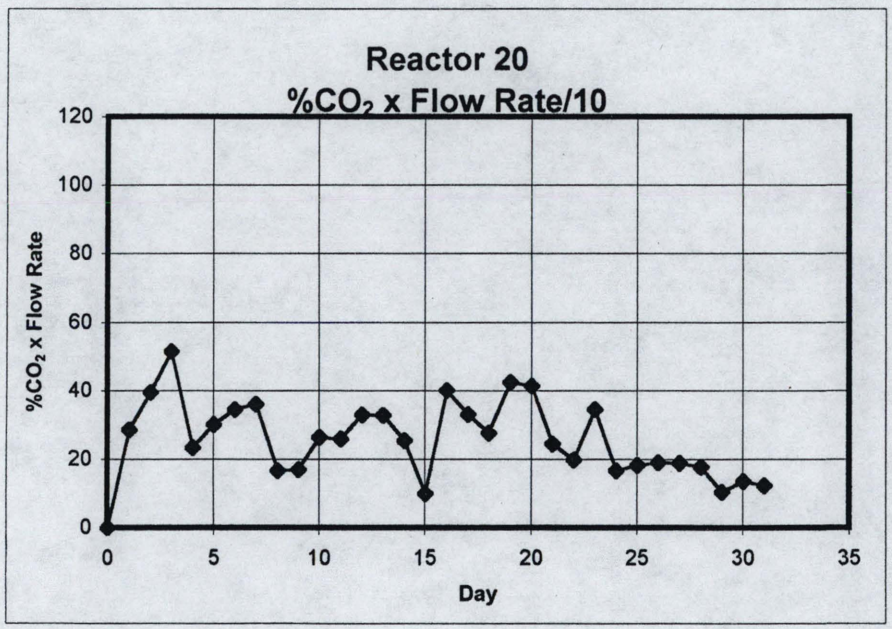

Reactor 26
%CO_2 x Flow Rate/10
%CO_2 x Flow Rate
Day
0
10
20
30
40
50
60
70
80

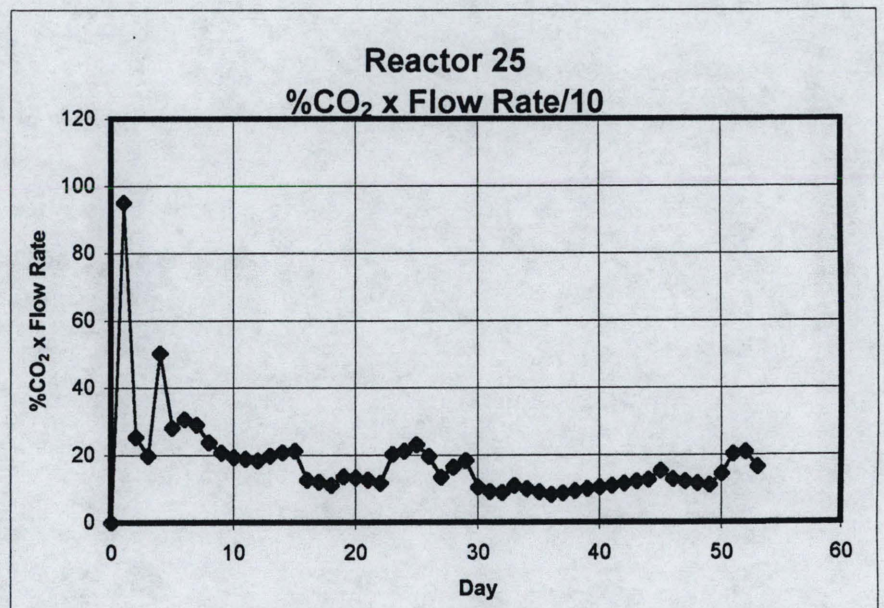

Reactor 18
%CO_2 x Flow Rate/10
%CO_2 x Flow Rate
Day
0
5
10
15
20
25
30
0
10
20
30
40
50
60
70
80
90
100


Reactor 24
%CO_2 x Flow Rate/10
%CO_2 x Flow Rate
Day
0
10
20
30
40
50
60
0
10
20
30
40
50
60
70
80
90
100


Reactor 19
%CO_2 x Flow Rate/10
%CO_2 x Flow Rate
Day


Reactor 23
%CO_2 x Flow Rate/10
%CO_2 x Flow Rate
Day


Reactor 20
%CO_2 x Flow Rate/10
%CO_2 x Flow Rate
Day


Reactor 25
%CO_2 x Flow Rate/10
%CO_2 x Flow Rate
Day

Summary of Results of Anaerobic Reactors **Table 3**

Reactor Numbers[1]	Gas Produced mmHg (x1000)	Methane %	Ash % t_o	Ash % t_f	% Converted Dry weight[2]	Total Days	Odors of Dried Reactants
A1	8.30	64.83	29.6	43.3	59.12	175	Fresh Barn Yard
A2	6.10	55.06	29.6	37.5	55.78	175	Fresh Barn Yard
A3	10.00	59.22	29.6	41.8	36.12	160	Fresh Dirt
A4	9.90	61.04	29.6	49.3	32.96	160	Fresh Dirt
A5	0.60	35.45	43.7	63.3	39.34	130	Fresh Barn Yard
A6	2.10	32.93	43.7	56.7	43.7	130	Fresh Barn Yard
A7	2.40	44.13	49.2	58.5	50.1	115	Foul/Sour Fermentation Smell
A8	0.90	0.01	45.6	51.9	14.56	53	Sharp Cheese/ Butyrates
A9	0.00	0.00	45.6	46.8	0	67	Fresh Dirt
A10	0.40	0.00	45.6	49.7	28.35	53	Sharp Cheese /Butyrates
A11	3.05	40.38	48.7	65.9	31.64	132	Earthy Smell
A12	1.67	0.08	53.4	41.3	28.11	132	Sharp Cheese/Butyrates
A13	0.87	3.95	ND	ND	ND	45**	Fermentation Smell
A14	0.08	0.00	52.5	52.4	0	47	Native Smell
A15	0.03	0.00	51.8	51.7	0	41	Foul Smell
A16	5.38	5.98	67.6	76.7	10.87	41	Fresh Dirt
A17	0.00	0.00	58.8	58.8	0	30	
A18	8.19	38.77	66.58	76.4	10.65	90	Earthy Smell/Fermentation
A19	9.96	43.92	54.45	TBD	TBD	74*	Earthy Smell/Fermentation
A20	9.80	46.79	54.45	TBD	TBD	74*	Earthy Smell/Fermentation
A21	10.25	66.59	54.45	TBD	TBD	74*	Earthy Smell/Fermentation

[1] See Table 1 and corresponding figures for gas profiles.

[2] Percentage of t_o and t_f dry weights of starting material and finished product, respectively.

* Still in operation; ** Stopped and used for seed culture; TBD to be determined; ND not determined

Calculation of Results of Anaerobic Reactors Table 4

Reactor Number[a]	Gas Produced in Liters[b]	Methane Liters[d]	Methane ft^3kg^{-1} [e]	BTU Kg^{-1} wet wt[e]	BTU Kg^{-1} dry wt	BTU Kg^{-1} TVS[h]	$Value per metric wet ton[g]
A1	49.8	32.3	0.72	704			3.31
A2	36.3	20	0.84	821			4.00
A3	60.0	35.5	2.22	2,152	4,304	6,114	10.00
A4	59.4	36.3	2.30	2,249	4,498	6,335	11.00
A5	31.0	1.1	0.07	69			0.32
A6	12.7	4.2	0.41	401			1.88
A7	14.3	6.3	0.61	597			2.81
A8	5.1	0	0	0			0.00
A9	<0.01	0	0	0			0.00
A10	2.4	0	0	0			0.00
A11	18.3	7.39	0.41	398			1.87
A12	10.0	0.79	NA	NA			NA
A13	5.2	0.2	NA	NA			NA
A14	0.5	0	NA	NA			NA
A15	0.2	0	NA	NA			NA
A16	32.3	1.9	0.03	34			0.16
A17	0	0	NA	NA			NA
A18	41	22.13	0.36	348	1,015	3,026	1.64
A19	39.8	17.48	0.65	637	1,490	3,276	2.99
A20	39.2	18.34	0.69	675	1,584	3,433	3.17
A21	46.3	30.83	1.08	1,056	2,396	5,281	4.96

*Reactors still in operation

[a]See Table 1 for identification of reaction mixtures

[b]Gas measured by displacement volume in graduated cylinders

[c]moles calculated from mm Hg, see corresponding reactor figures

[d]Percentage determined by Gas Chromatographic profile, see reactor figures of mole-%

[e]One Liter = 0.035 ft^3; Kg represent wet weight of 35-50% water and ash content of 20-68%

[f]978Btu/ft^3

[g]100,000 Btu's = $0.47, approximately.

[h]TVS = Total Volatile Solids

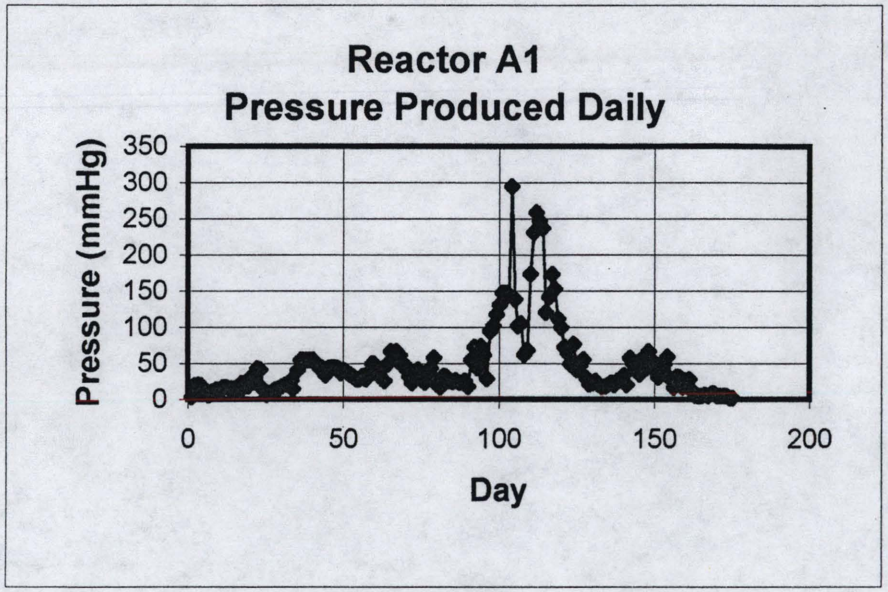
Nitrogen, Carbon and Hydrogen Analyses **Table 5**

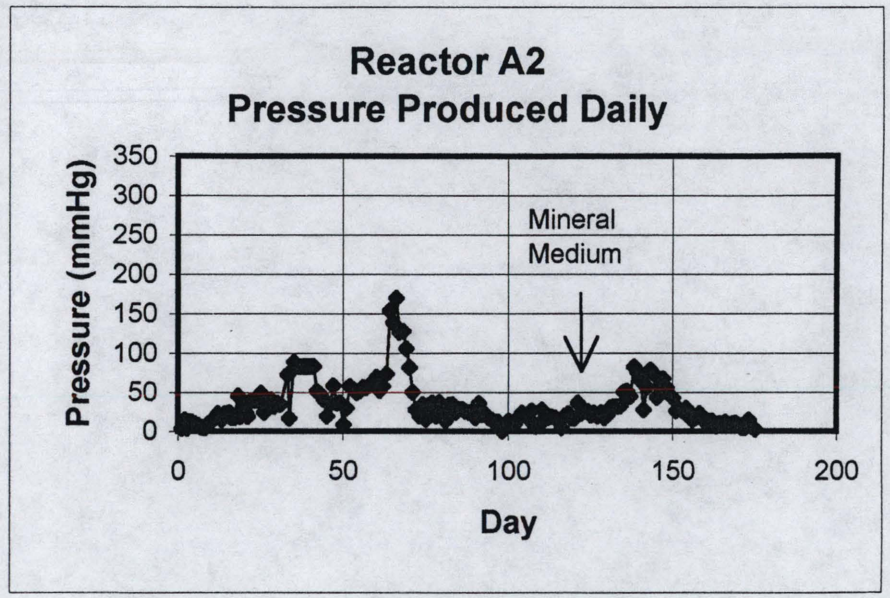
Sample	ug N	%N	ug C	% C	ug H	% H	g N added	g Final N	% Remaining
A1	38.08	0.64	1968.09	33.17	205.91	3.47	10.6	12.08	114
A2	51.36	0.57	3057.69	33.99	346.9	3.86	8.11	5.45	67.2
A3	60.72	0.74	2829.09	34.28	316.5	3.83	6.69	4.78	71.5
A4	75.87	0.89	2805.21	32.84	320.15	3.75	7.05	5.55	78.7
A5	86.34	1.3	1773.45	26.76	203.08	3.06	8	7.94	99.2
A6	67.88	0.92	2105.18	28.38	236.83	3.19	8	8	100
A8	31.95	0.58	1583.45	28.95	170.85	3.12	8	8	100

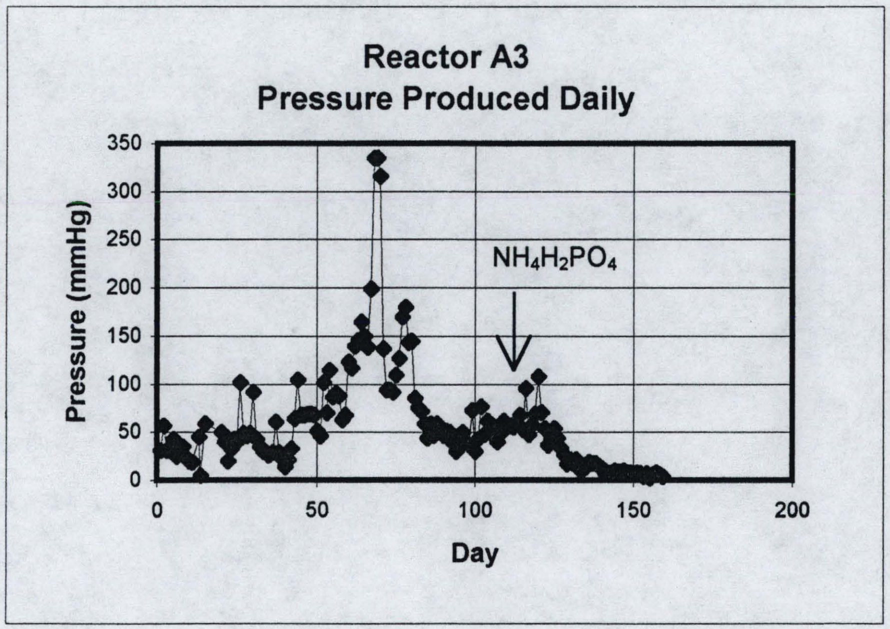
Organism Counts

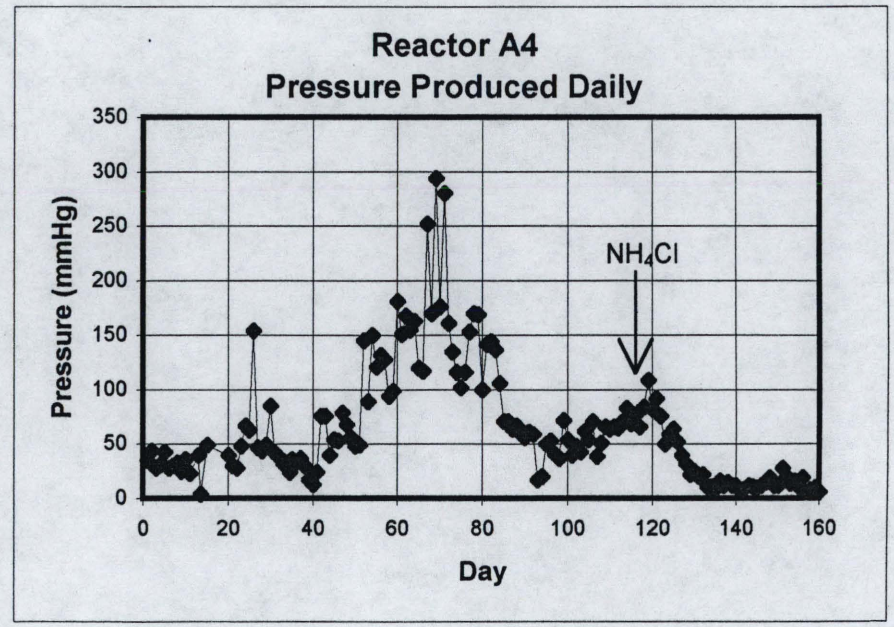
Table 6

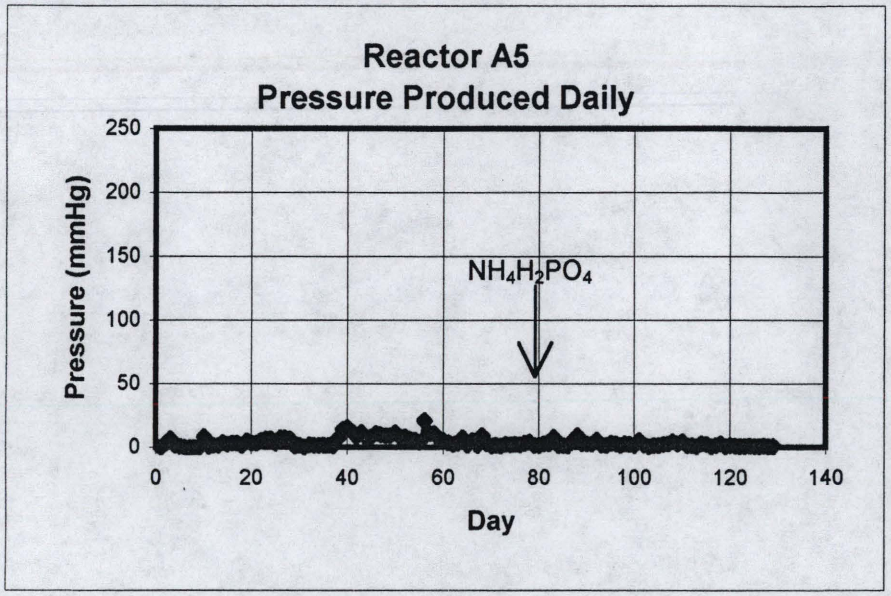
T_0	1/10 TSA (organisms/g)	1/50 TSA (organisms/g)	Potato Dextrose with Streptomycin (organisms/g)	Potato Dextrose with Ampicillin (organisms/g)
Reactor 8	TNTC	5.7E+3	18.0E+3	9.8E+3
Reactor 10	TNTC	5.7E+3	18.0E+3	9.8E+3
Reactor 11	TNTC	5.7E+3	18.0E+3	9.8E+3
Reactor 12	TNTC	5.7E+3	18.0E+3	9.8E+3
T_{12}				
Reactor 8	2.8E+11	2.7E+11	1E+11	TNTC
Reactor 10	2.4E+11	2E+11	1.4E+11	2.0E+9
Reactor 11	1.8E+11	1.7E+11	1.5E+11	4.1E+9
Reactor 12	1.6E+11	1.3E+11	1.2E+11	3.7E+9
T_{31}				
Reactor 8	1.9E+9	2.2E+9	2.90E+08	5.10E+08
Reactor 10	2.4E+9	2.6E+9	2.80E+08	2.10E+08
Reactor 11	2.70E+10	2.50E+10	1.4E+9	9.90E+08
Reactor 12	3.30E+10	3.20E+10	3.5E+9	1.6E+9

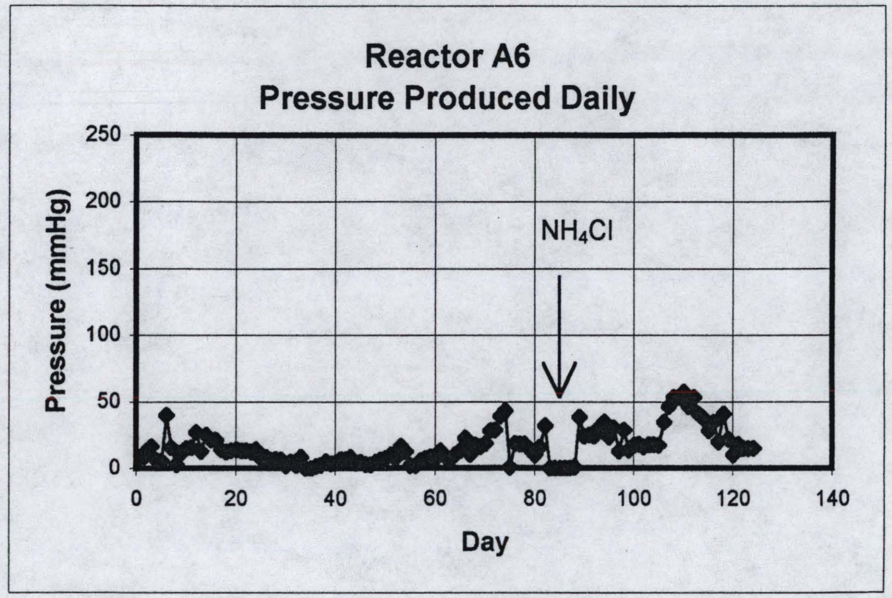

Reactor A1
Pressure Produced Daily
Pressure (mmHg)
Day

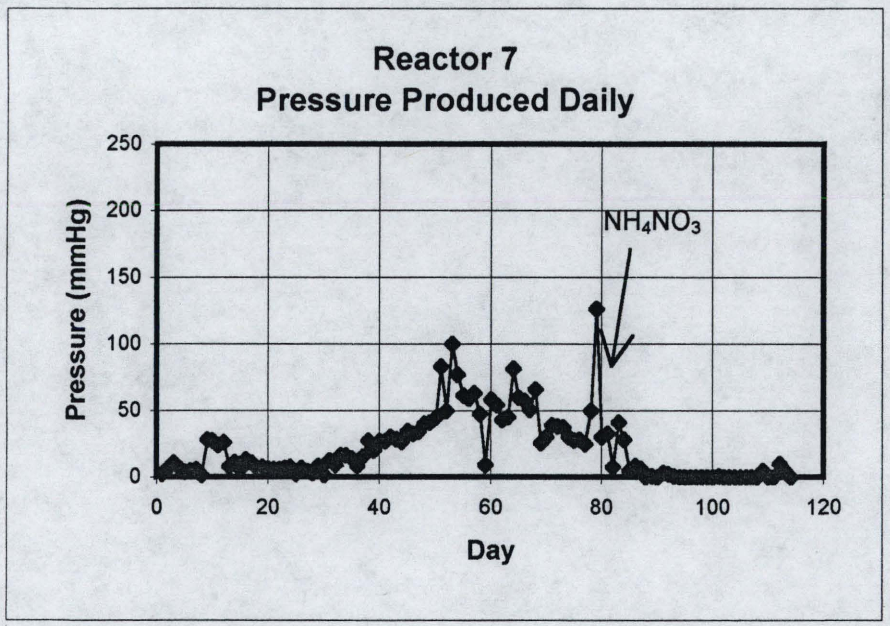

Reactor A2
Pressure Produced Daily
Pressure (mmHg)
Day
Mineral Medium

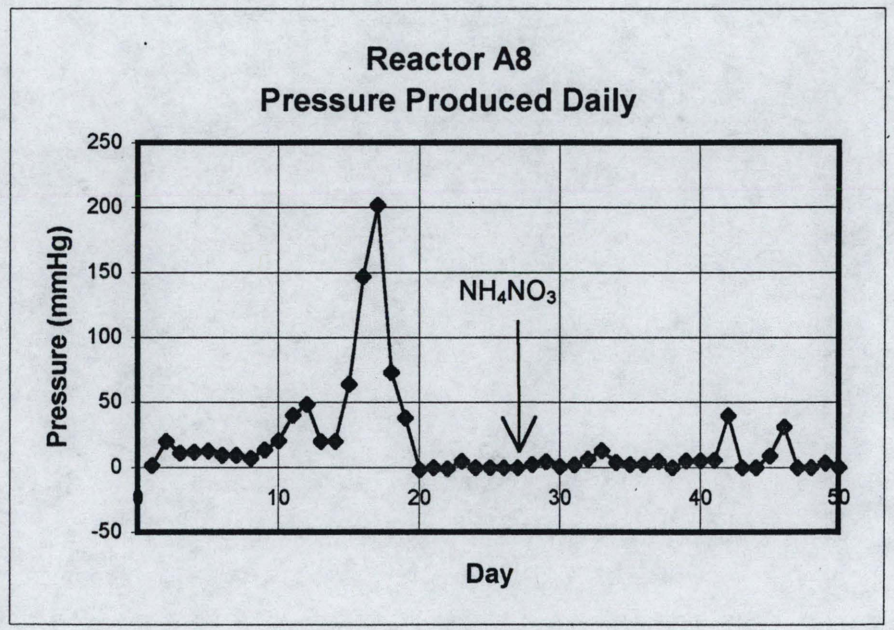

Reactor A3
Pressure Produced Daily
Pressure (mmHg)
Day
$NH_4H_2PO_4$

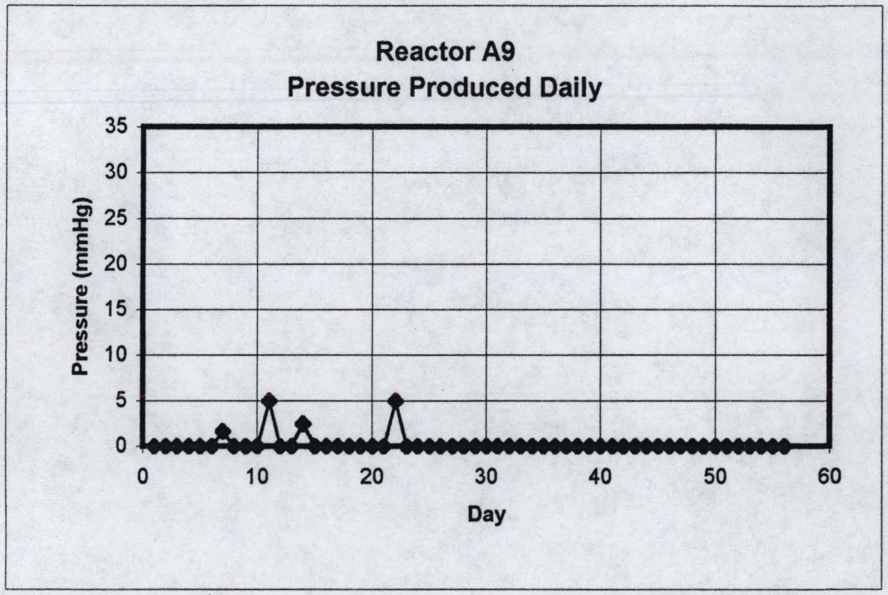

Reactor A4
Pressure Produced Daily
Pressure (mmHg)
Day
NH_4Cl


Reactor A5
Pressure Produced Daily
Pressure (mmHg)
Day
NH4H2PO4
250
200
150
100
50
0
0
20
40
60
80
100
120
140

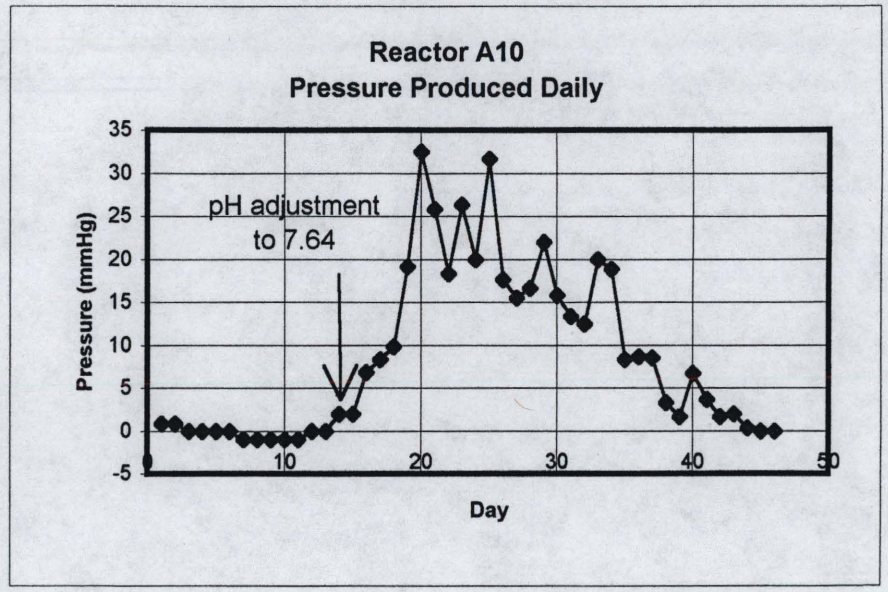

Reactor A6
Pressure Produced Daily
Pressure (mmHg)
Day
NH4Cl
250
200
150
100
50
0
0
20
40
60
80
100
120
140


Reactor 7
Pressure Produced Daily
Pressure (mmHg)
Day
NH4NO3
250
200
150
100
50
0
0
20
40
60
80
100
120

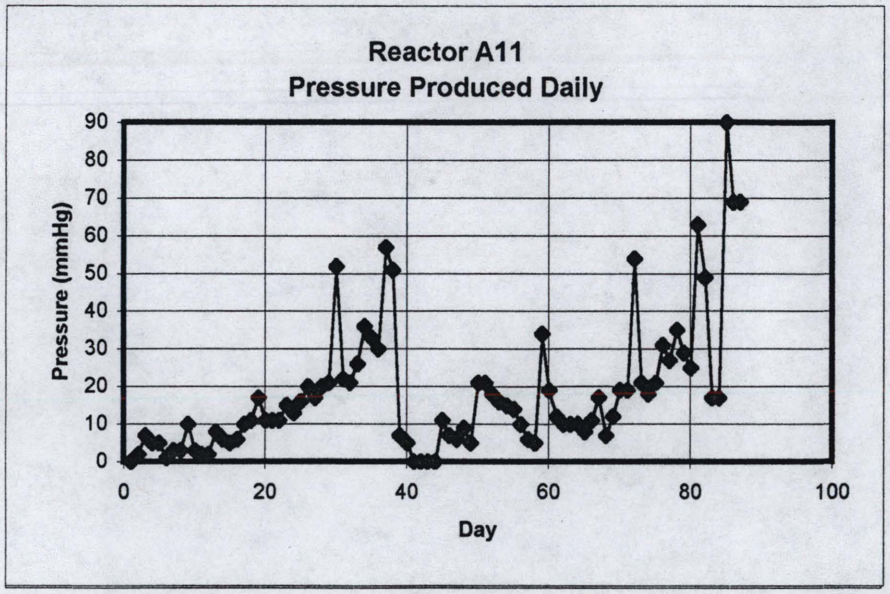

Reactor A8
Pressure Produced Daily
Pressure (mmHg)
Day
NH4NO3
250
200
150
100
50
0
-50
10
20
30
40
50

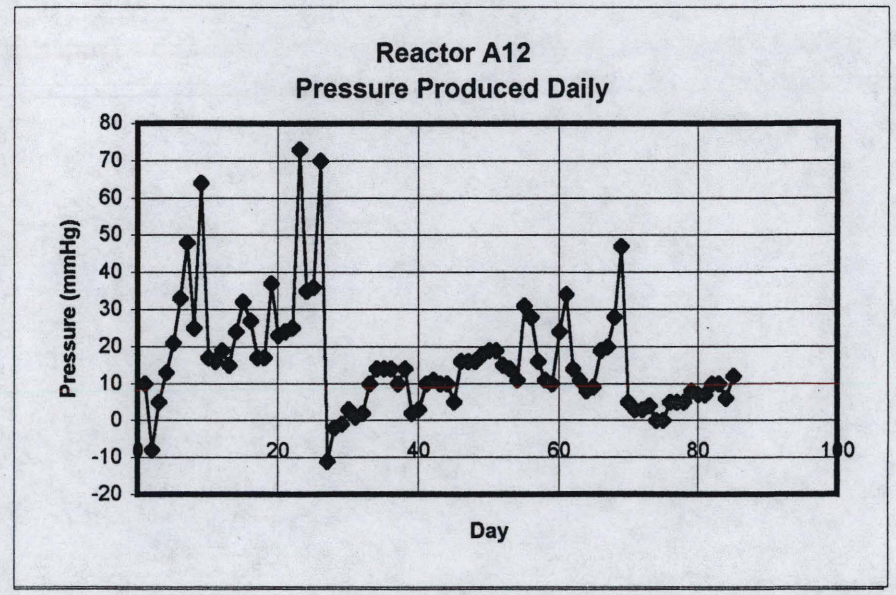


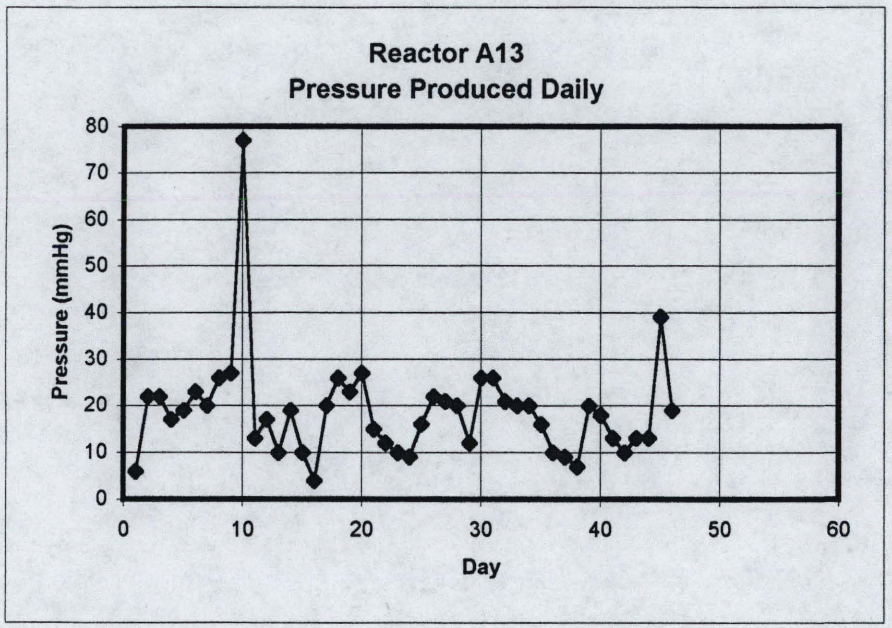
Reactor A9
Pressure Produced Daily
Pressure (mmHg)
Day

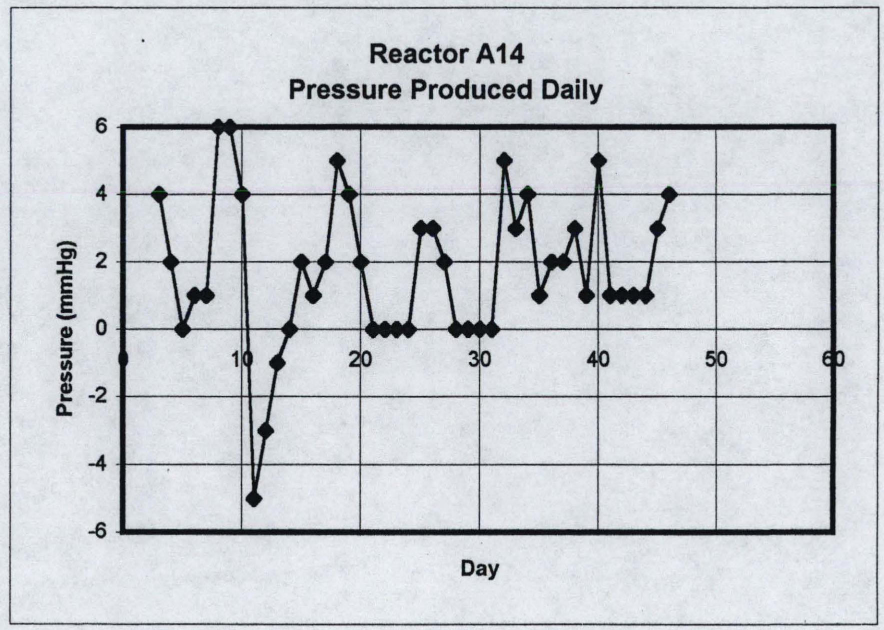


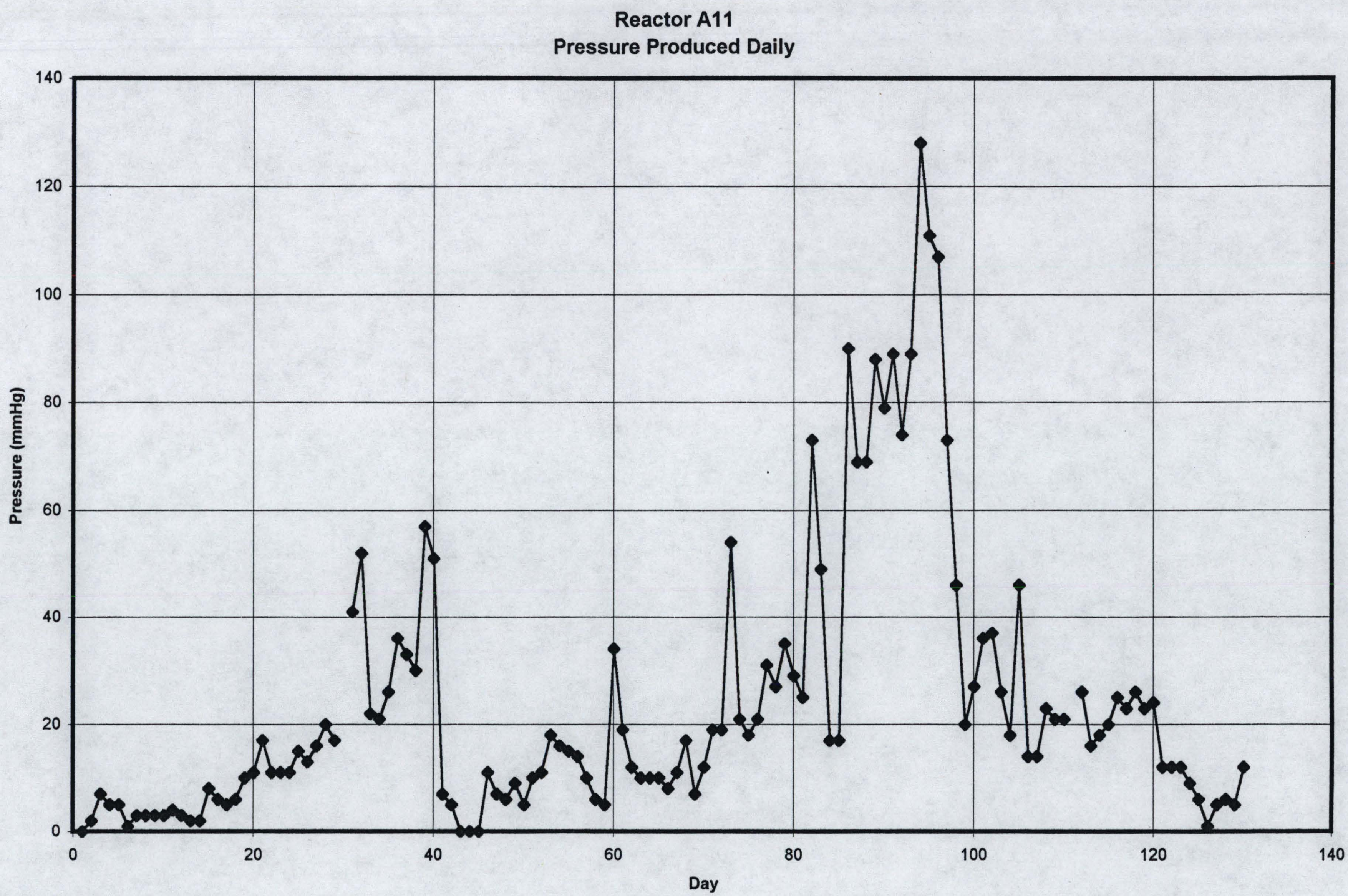
Reactor A10
Pressure Produced Daily
pH adjustment to 7.64
Pressure (mmHg)
Day


Reactor A11
Pressure Produced Daily
Pressure (mmHg)
Day
0
10
20
30
40
50
60
70
80
90
0
20
40
60
80
100


Reactor A12
Pressure Produced Daily
Pressure (mmHg)
Day
-20
-10
0
10
20
30
40
50
60
70
80
20
40
60
80
100

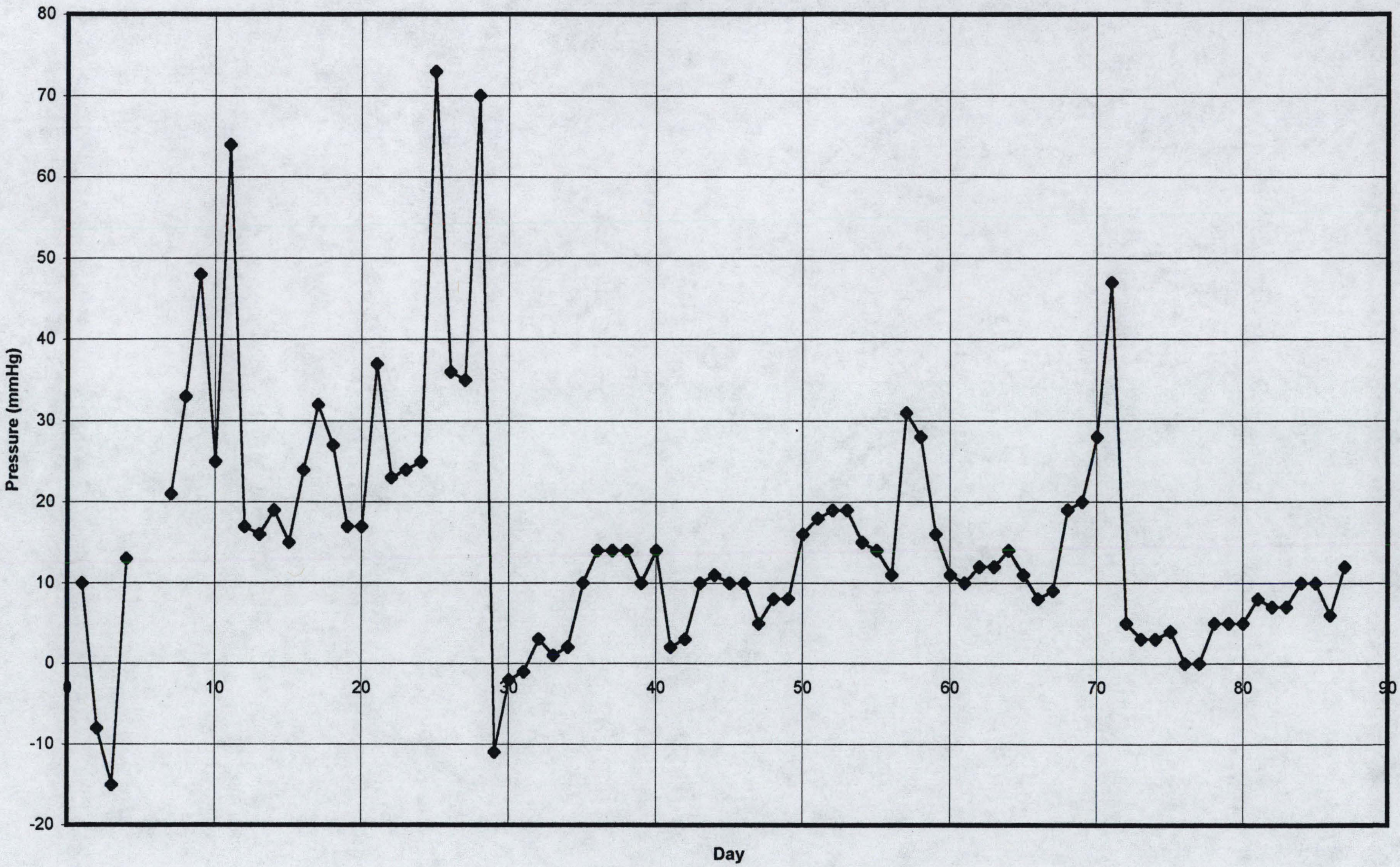

Reactor A13
Pressure Produced Daily
Pressure (mmHg)
Day
0
10
20
30
40
50
60
70
80
0
10
20
30
40
50
60


Reactor A14
Pressure Produced Daily
Pressure (mmHg)
Day
-6
-4
-2
0
2
4
6
10
20
30
40
50
60

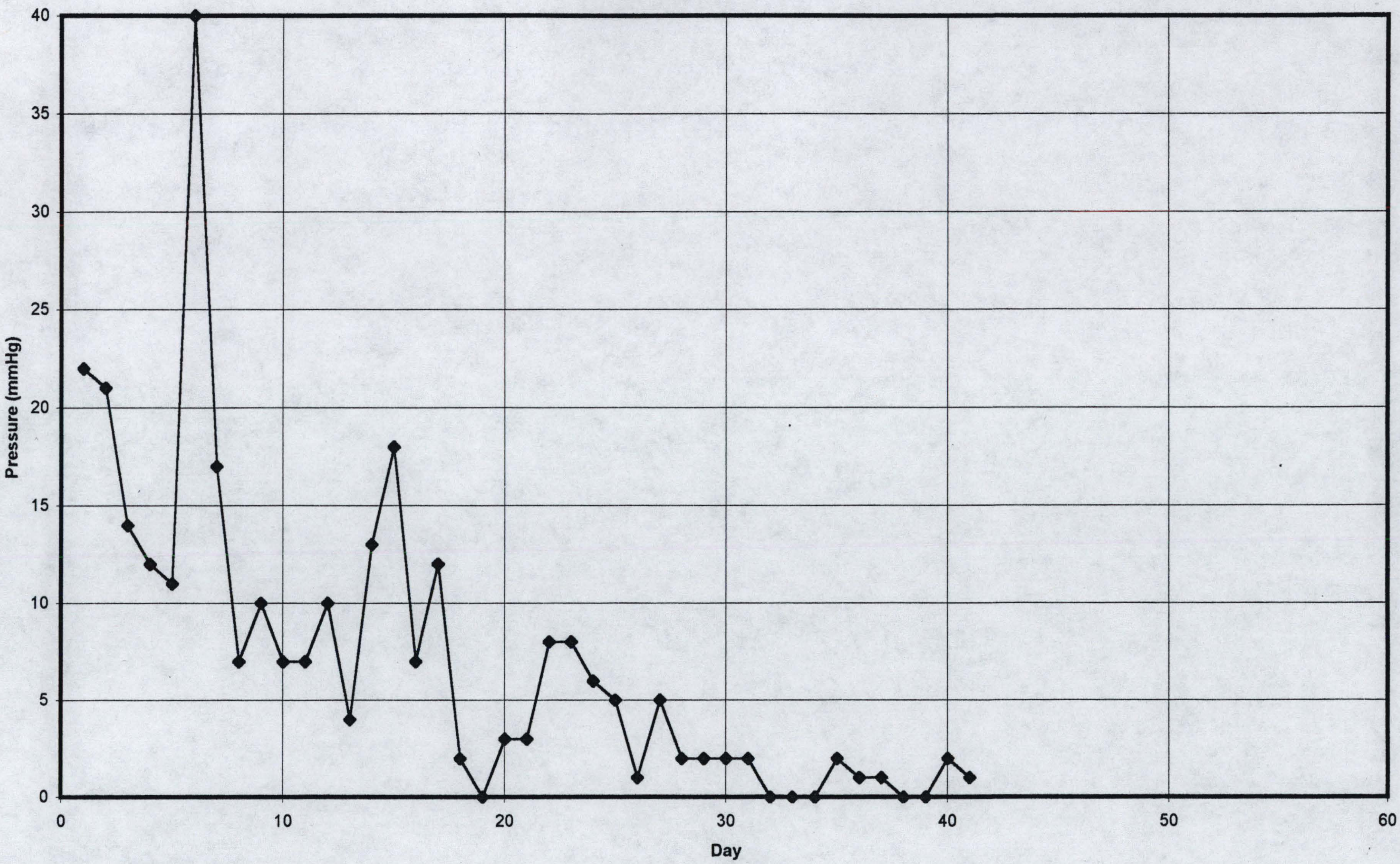


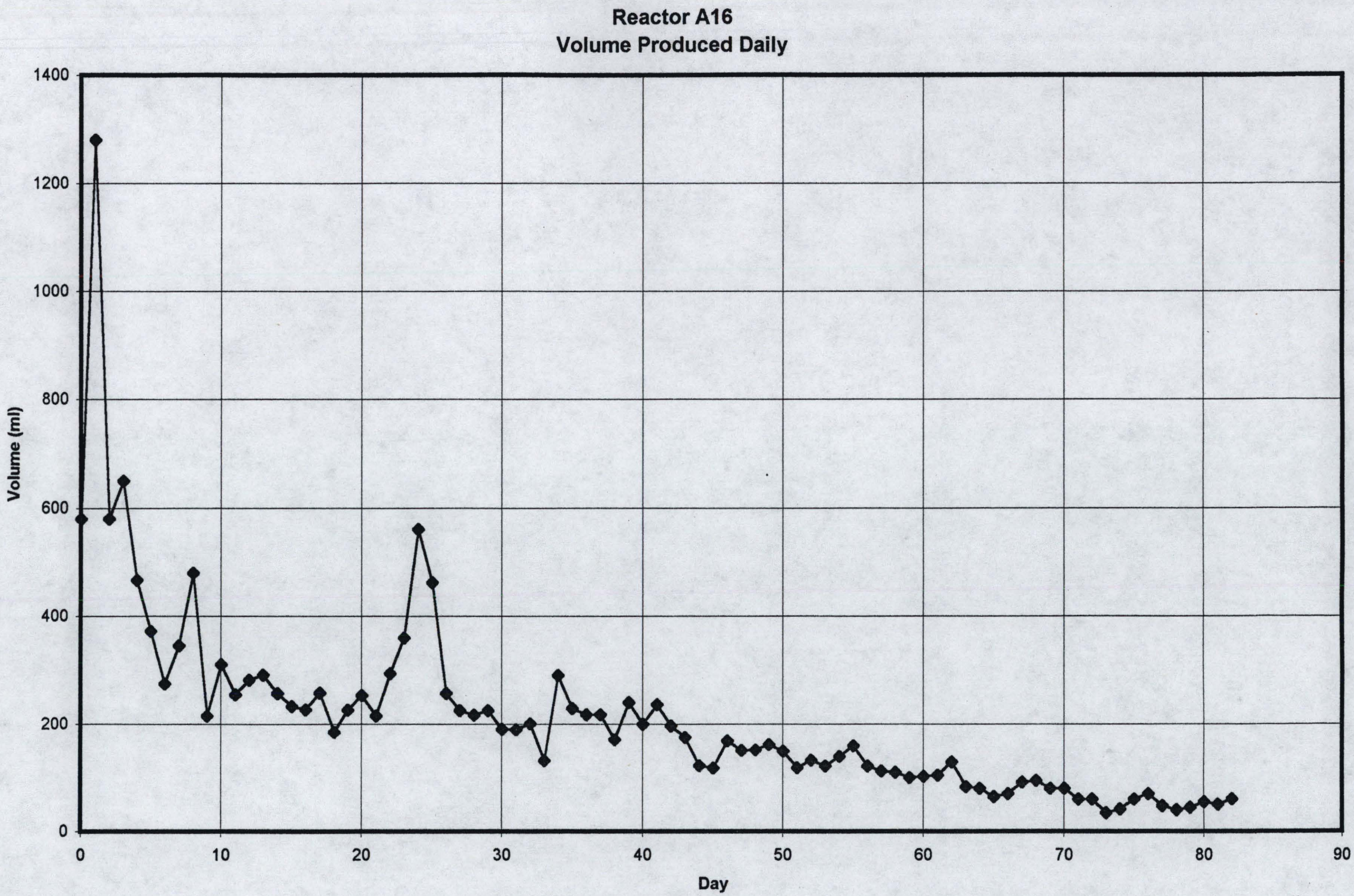
Reactor A11
Pressure Produced Daily
Pressure (mmHg)
140
120
100
80
60
40
20
0
0
20
40
60
80
100
120
140
Day

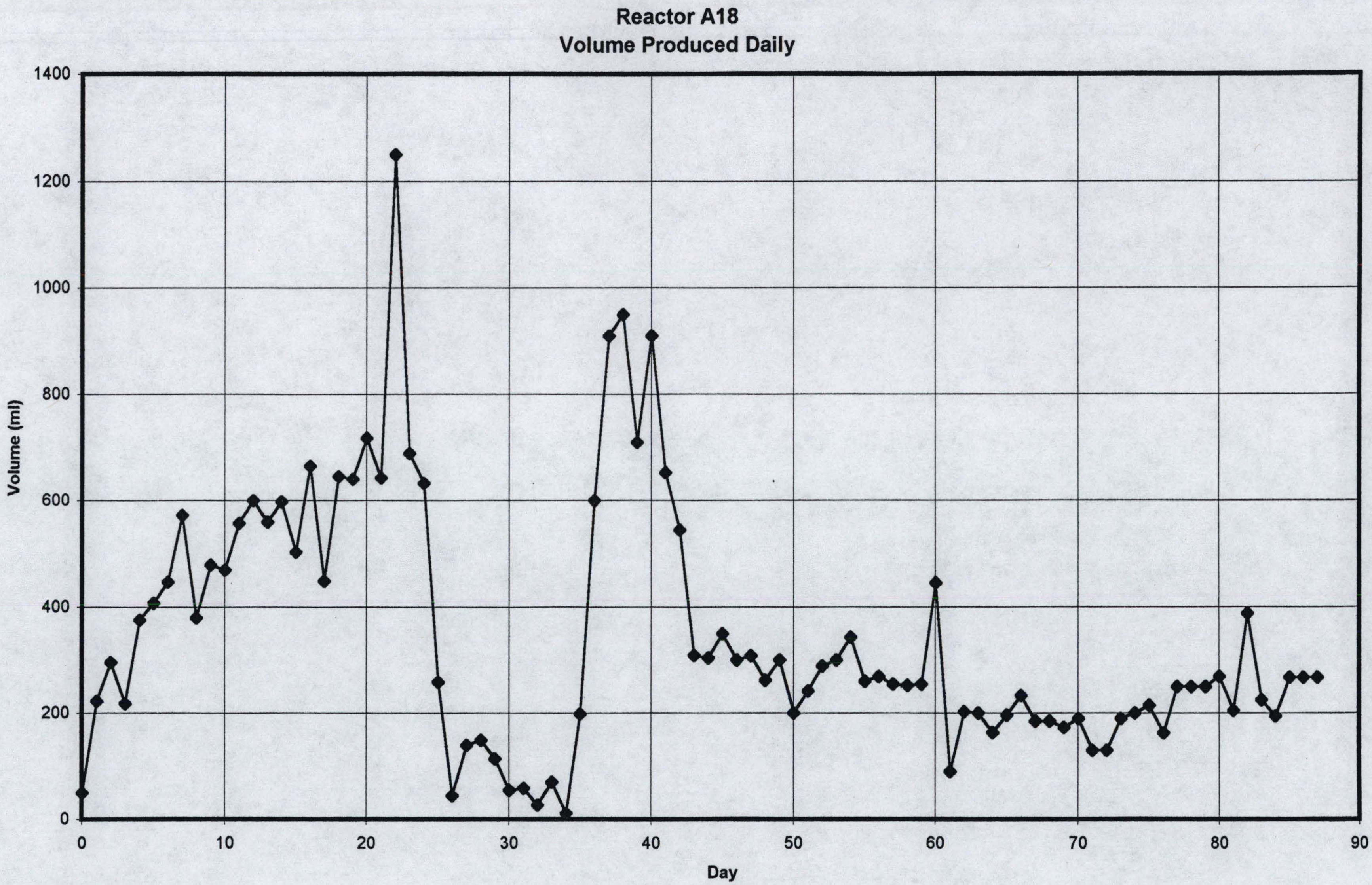
Reactor A12
Pressure Produced Daily



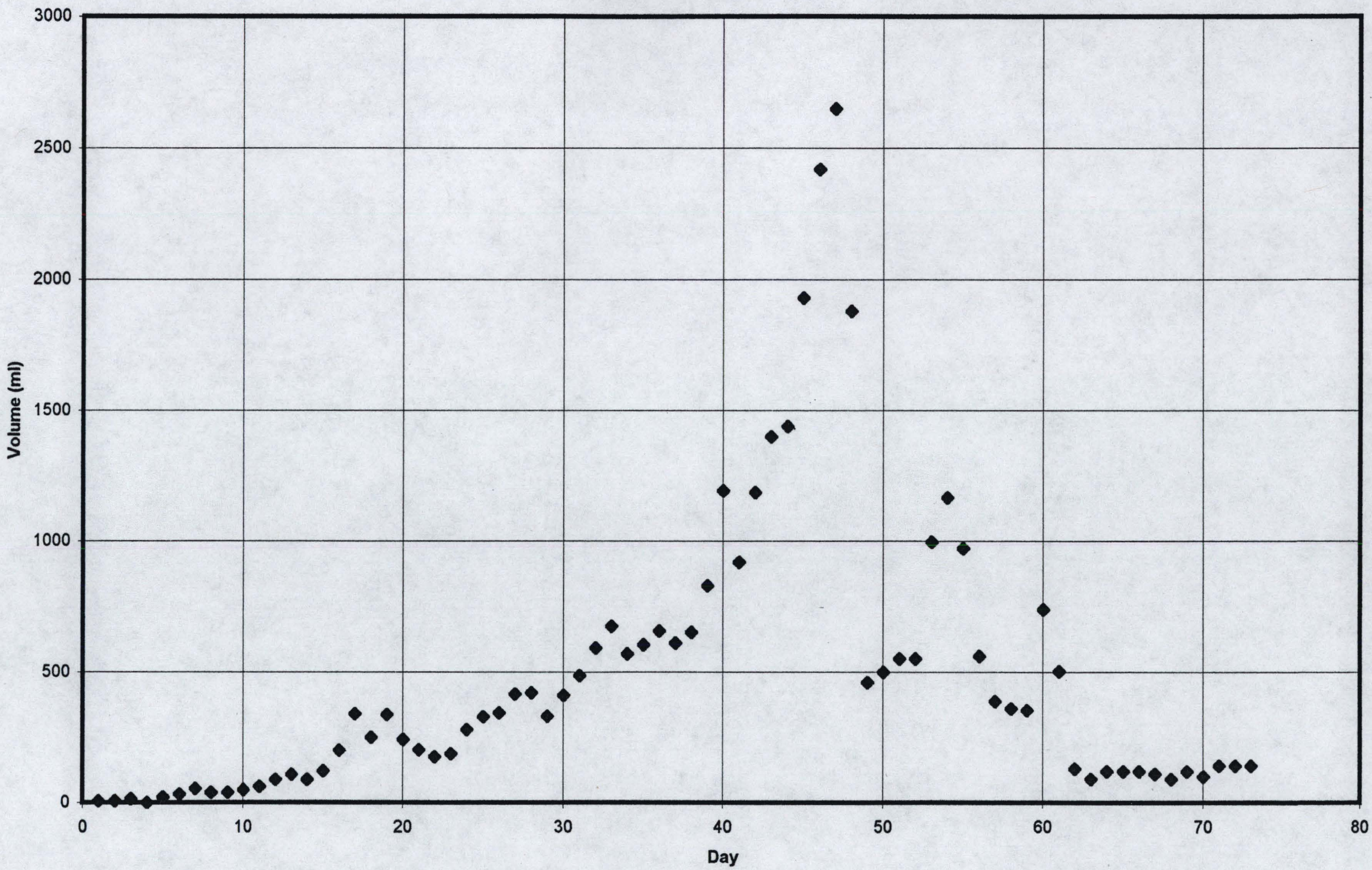
Reactor A15
Pressure Produced Daily



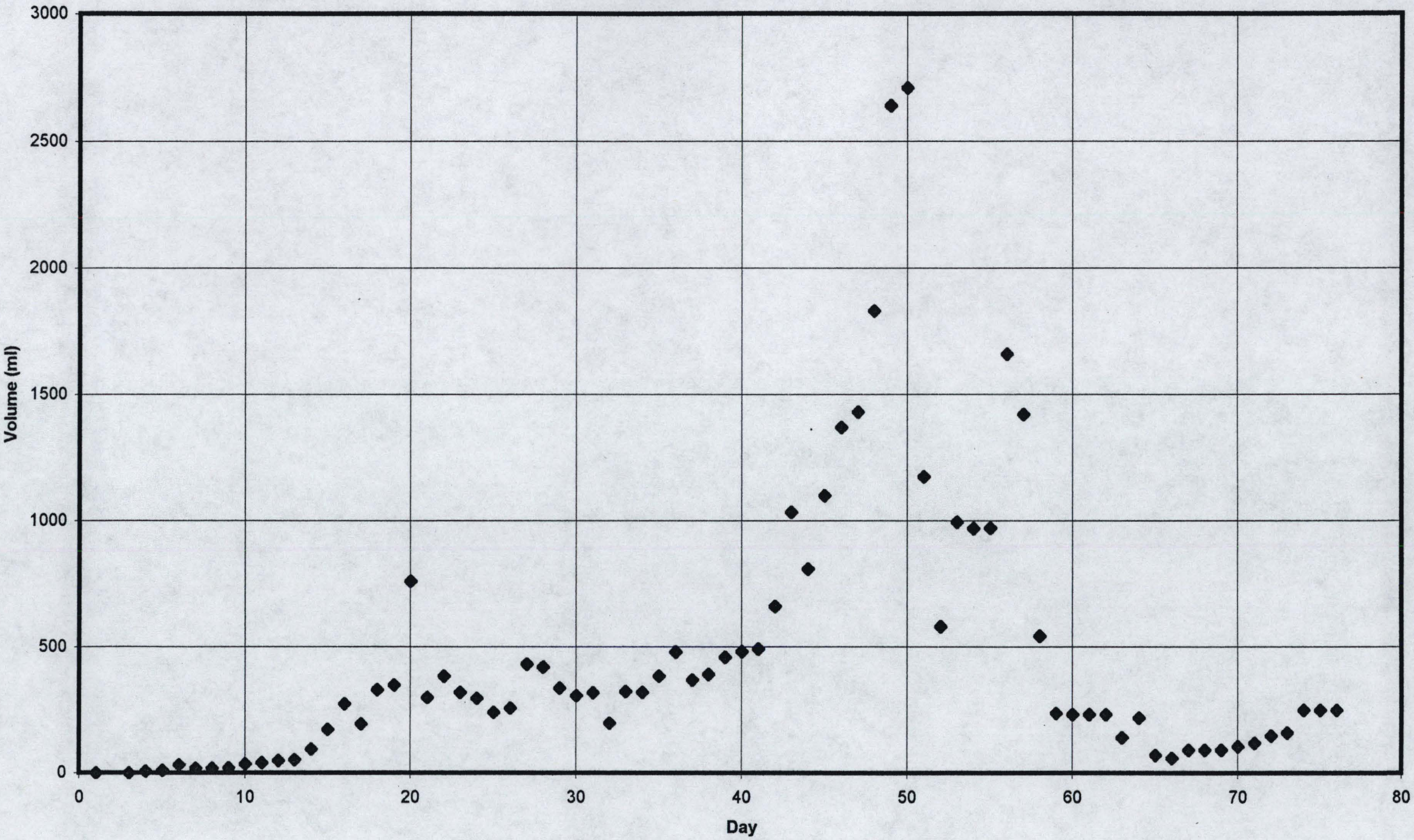

Reactor A16
Volume Produced Daily
Volume (ml)
Day
0
200
400
600
800
1000
1200
1400
10
20
30
40
50
60
70
80
90

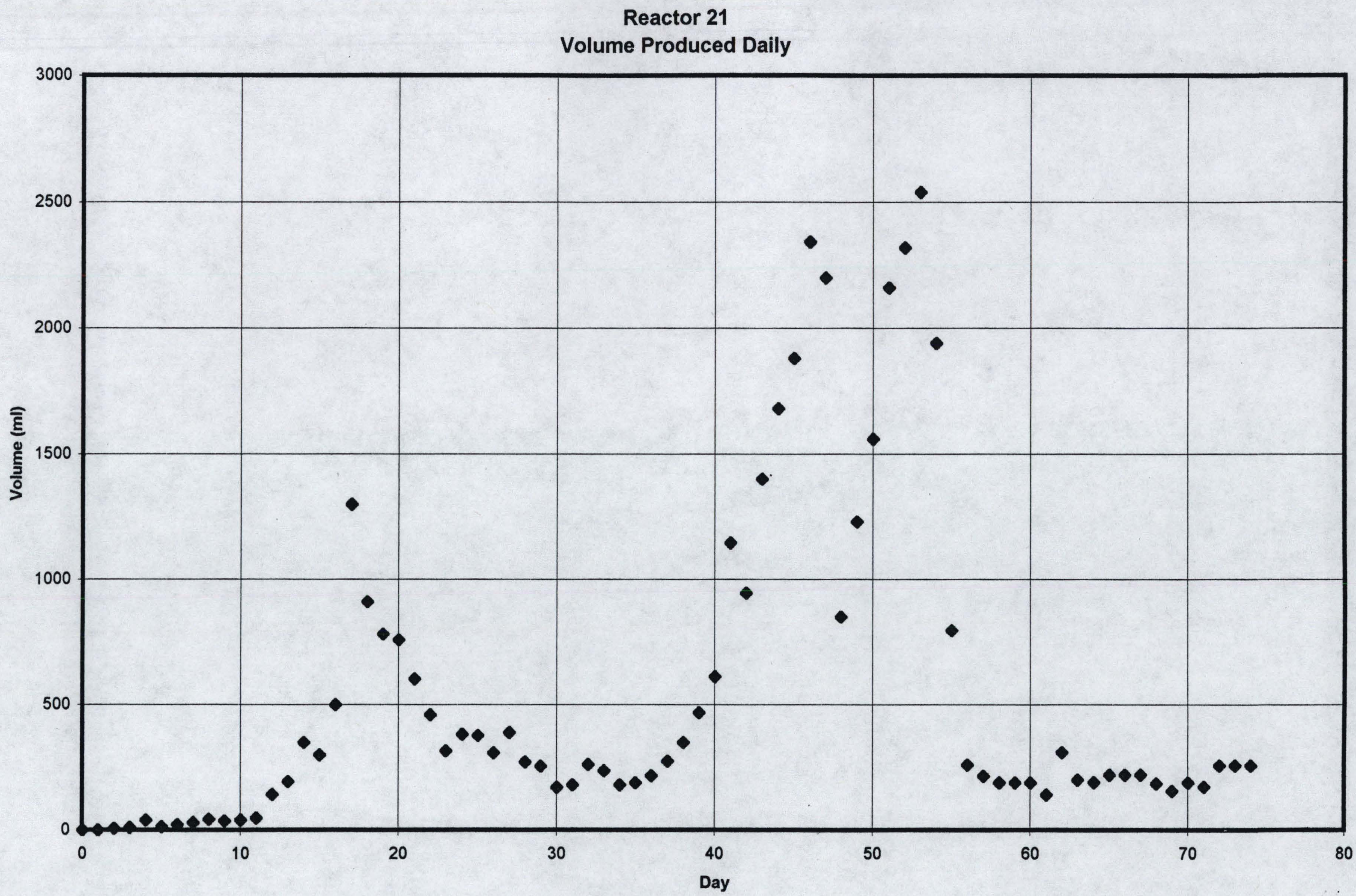

Reactor A18
Volume Produced Daily
Volume (ml)
Day
0
200
400
600
800
1000
1200
1400
0
10
20
30
40
50
60
70
80
90

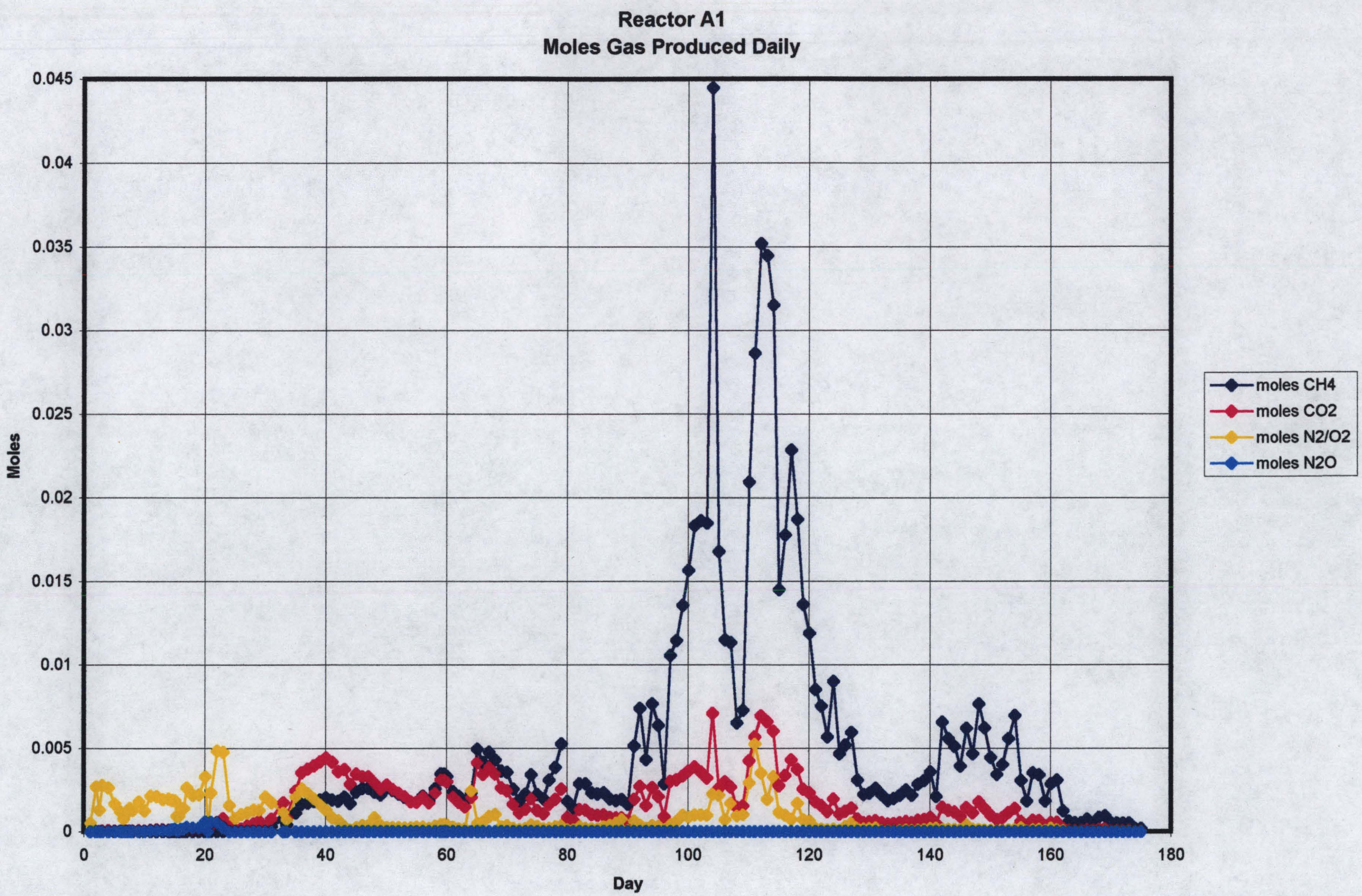
Reactor A19
Volume Produced Daily



Reactor A20
Volume Produced Daily

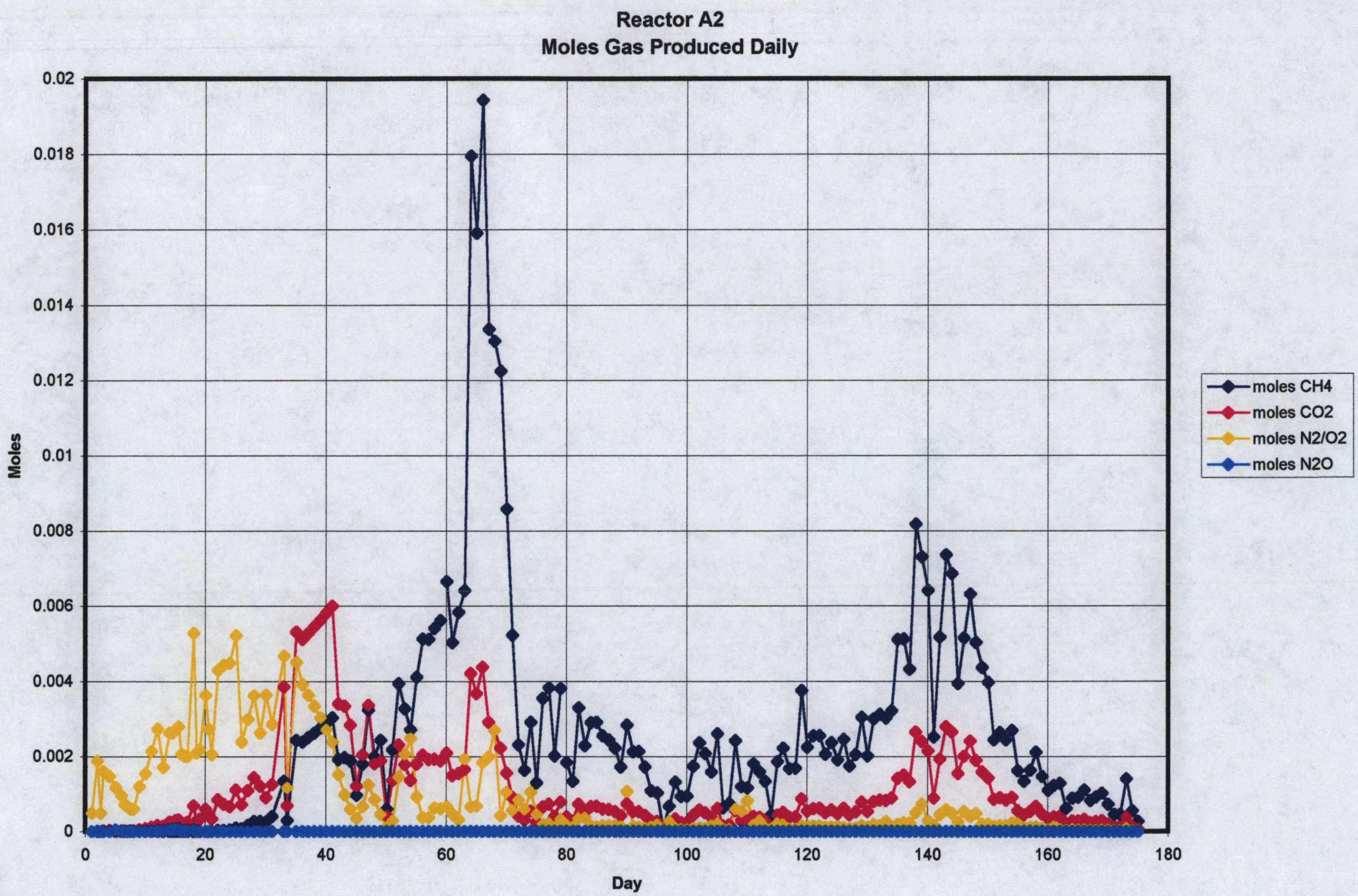



Reactor 21
Volume Produced Daily
Volume (ml)
Day
3000
2500
2000
1500
1000
500
0
0
10
20
30
40
50
60
70
80



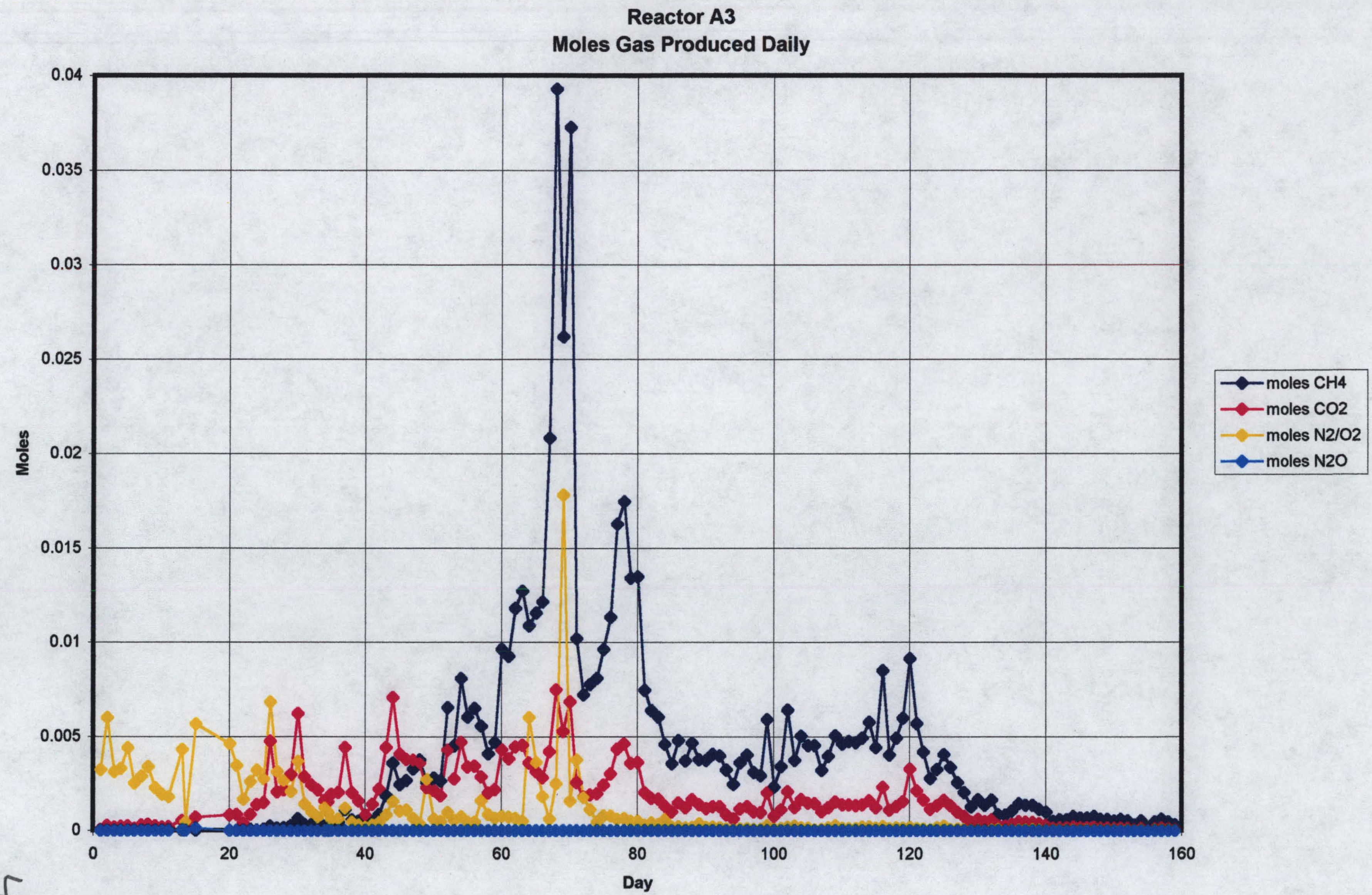
Reactor A1
Moles Gas Produced Daily
0.045
0.04
0.035
0.03
0.025
0.02
0.015
0.01
0.005
0
Moles
0
20
40
60
80
100
120
140
160
180
Day
moles CH4
moles CO2
moles N2/O2
moles N2O

47



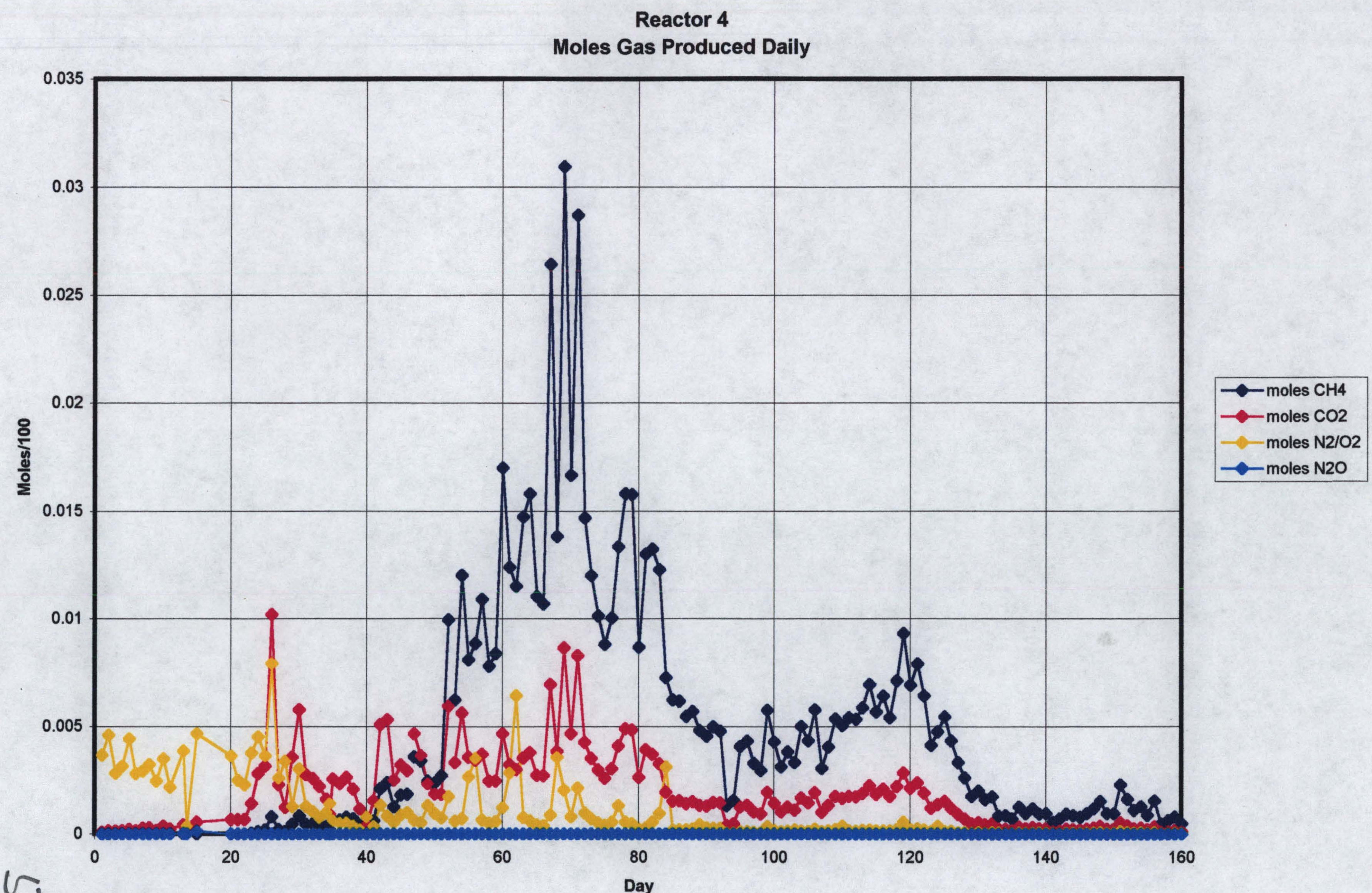
Reactor A2
Moles Gas Produced Daily
Moles
0.02
0.018
0.016
0.014
0.012
0.01
0.008
0.006
0.004
0.002
0
0
20
40
60
80
100
120
140
160
180
Day
moles CH4
moles CO2
moles N2/O2
moles N2O

48



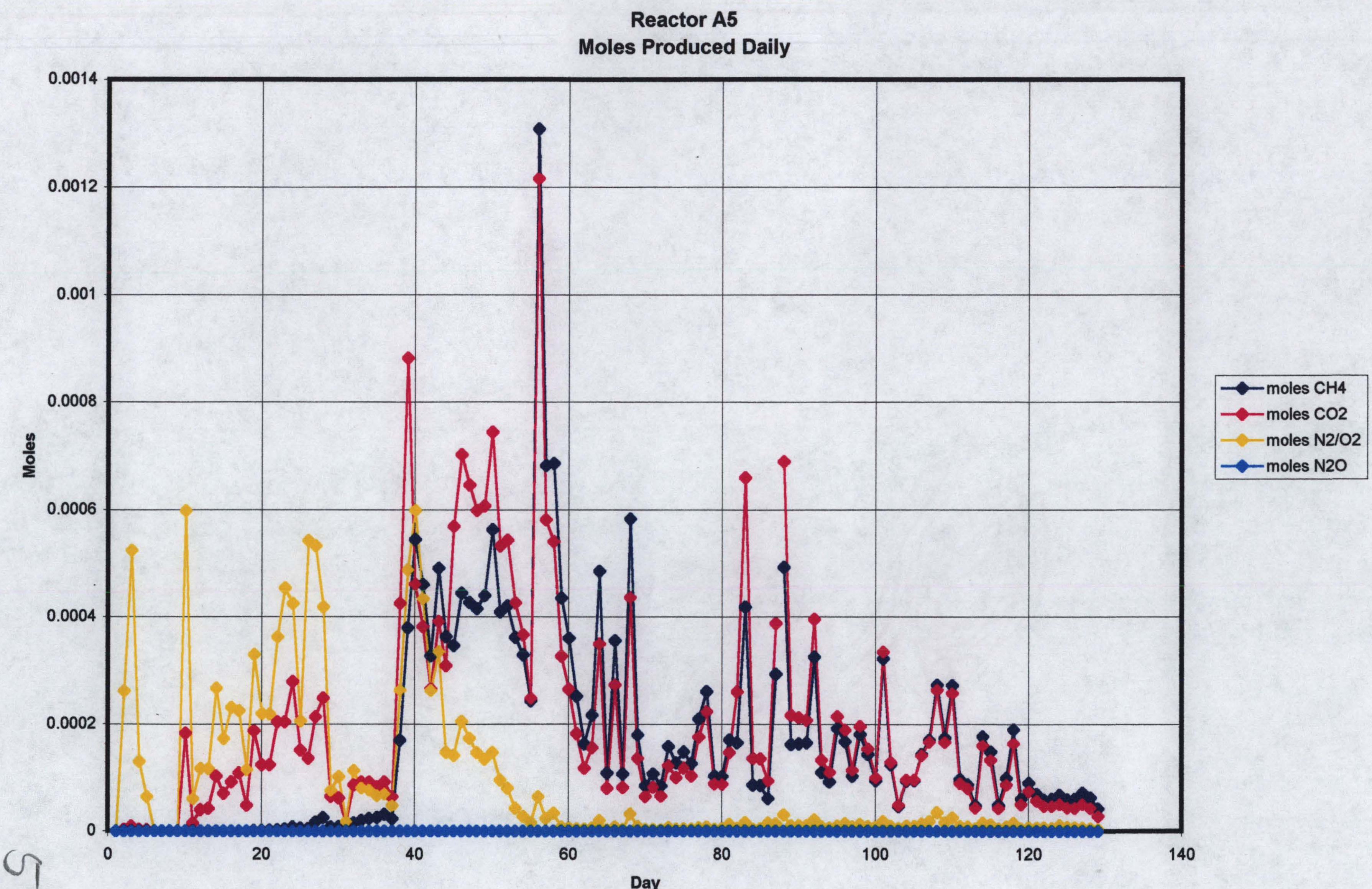
Reactor A3
Moles Gas Produced Daily
Moles
0.04
0.035
0.03
0.025
0.02
0.015
0.01
0.005
0
0
20
40
60
80
100
120
140
160
Day
moles CH4
moles CO2
moles N2/O2
moles N2O

49



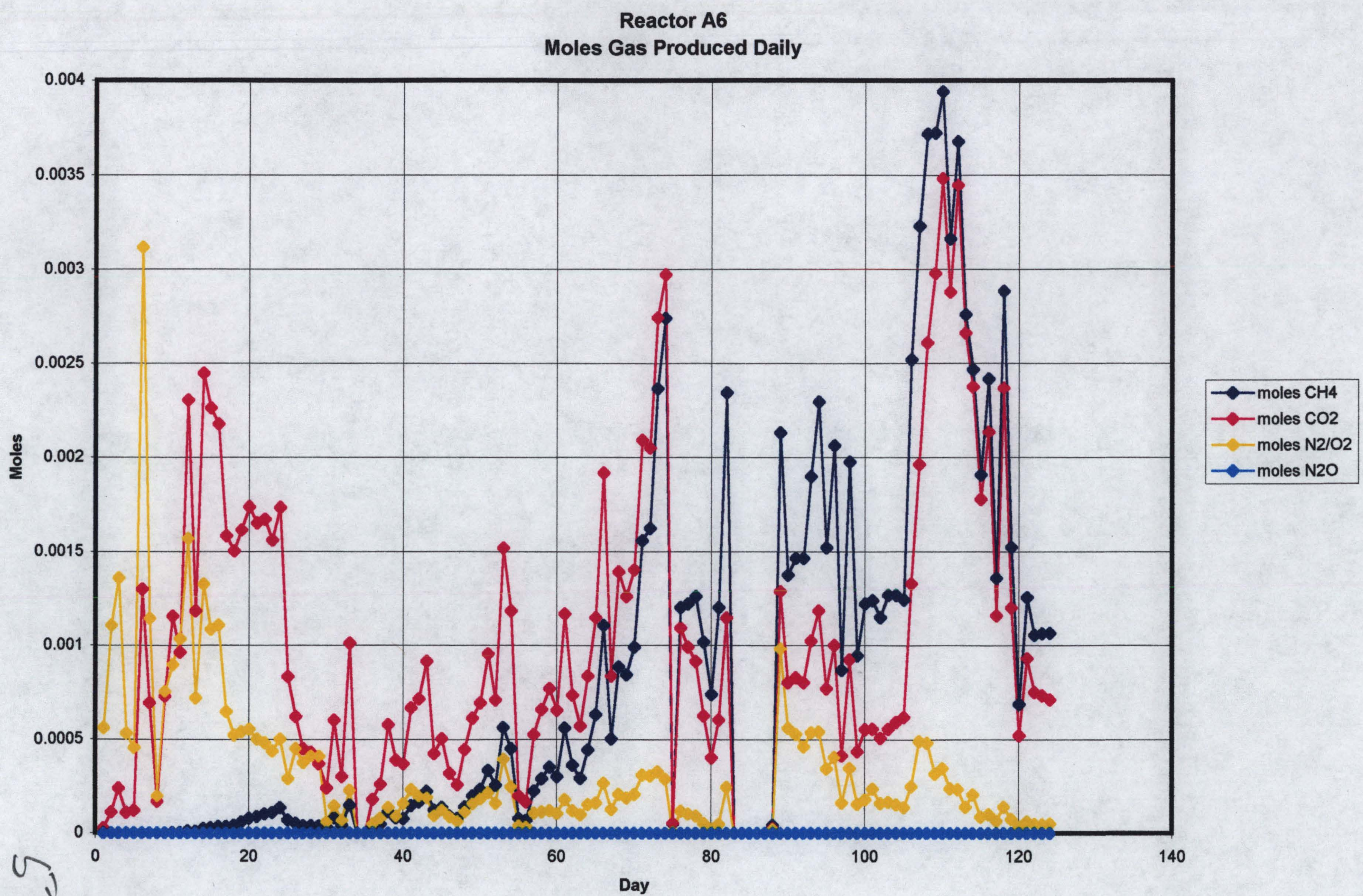
Reactor 4
Moles Gas Produced Daily
Moles/100
0.035
0.03
0.025
0.02
0.015
0.01
0.005
0
0
20
40
60
80
100
120
140
160
Day
moles CH4
moles CO2
moles N2/O2
moles N2O

55



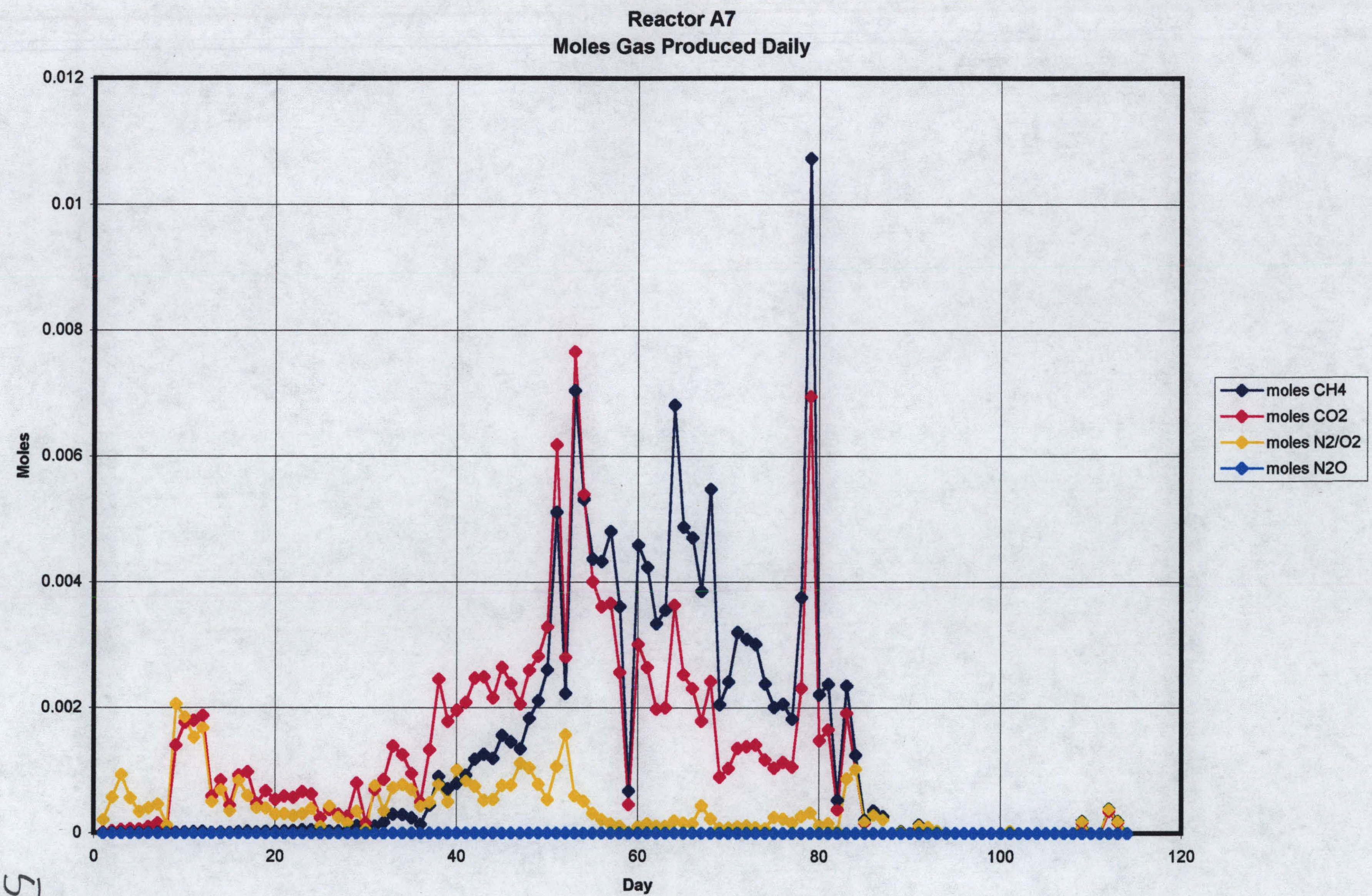
Reactor A5
Moles Produced Daily
0.0014
0.0012
0.001
0.0008
0.0006
0.0004
0.0002
0
Moles
0
20
40
60
80
100
120
140
Day
moles CH4
moles CO2
moles N2/O2
moles N2O

5



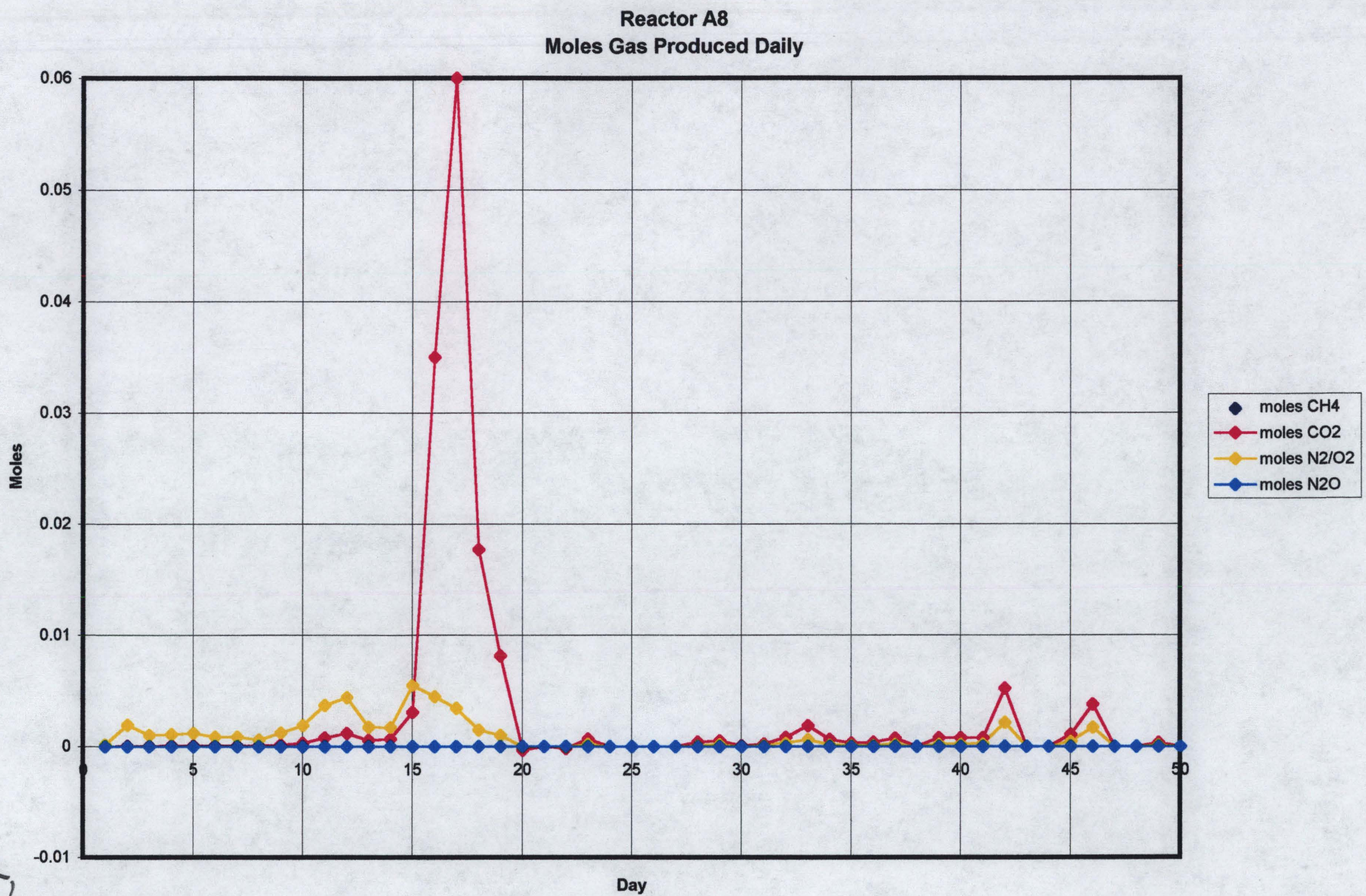
Reactor A6
Moles Gas Produced Daily
0.004
0.0035
0.003
0.0025
0.002
0.0015
0.001
0.0005
0
Moles
0
20
40
60
80
100
120
140
Day
moles CH4
moles CO2
moles N2/O2
moles N2O

25



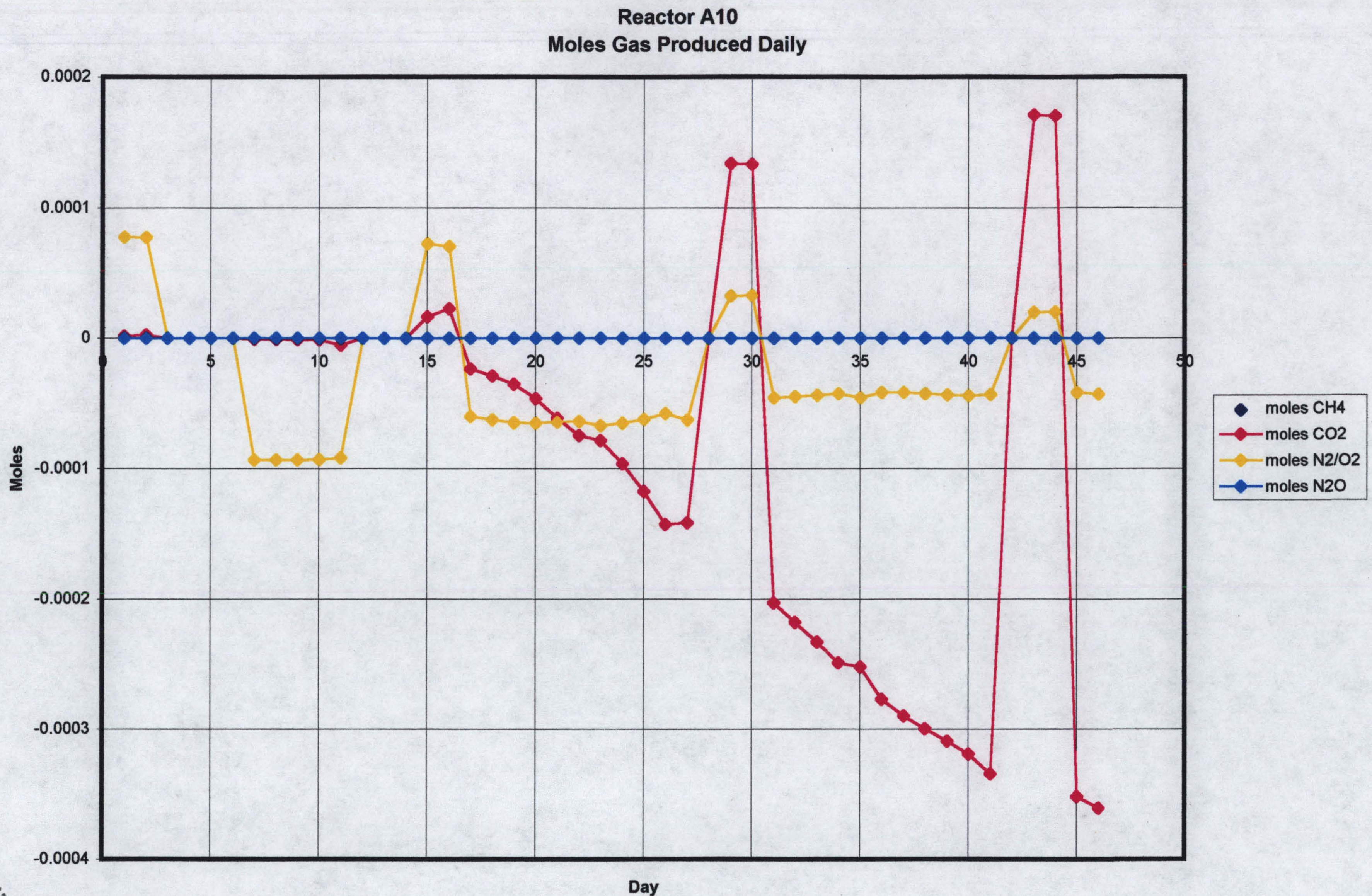
Reactor A7
Moles Gas Produced Daily
0.012
0.01
0.008
0.006
0.004
0.002
0
Moles
0
20
40
60
80
100
120
Day
moles CH4
moles CO2
moles N2/O2
moles N2O

63



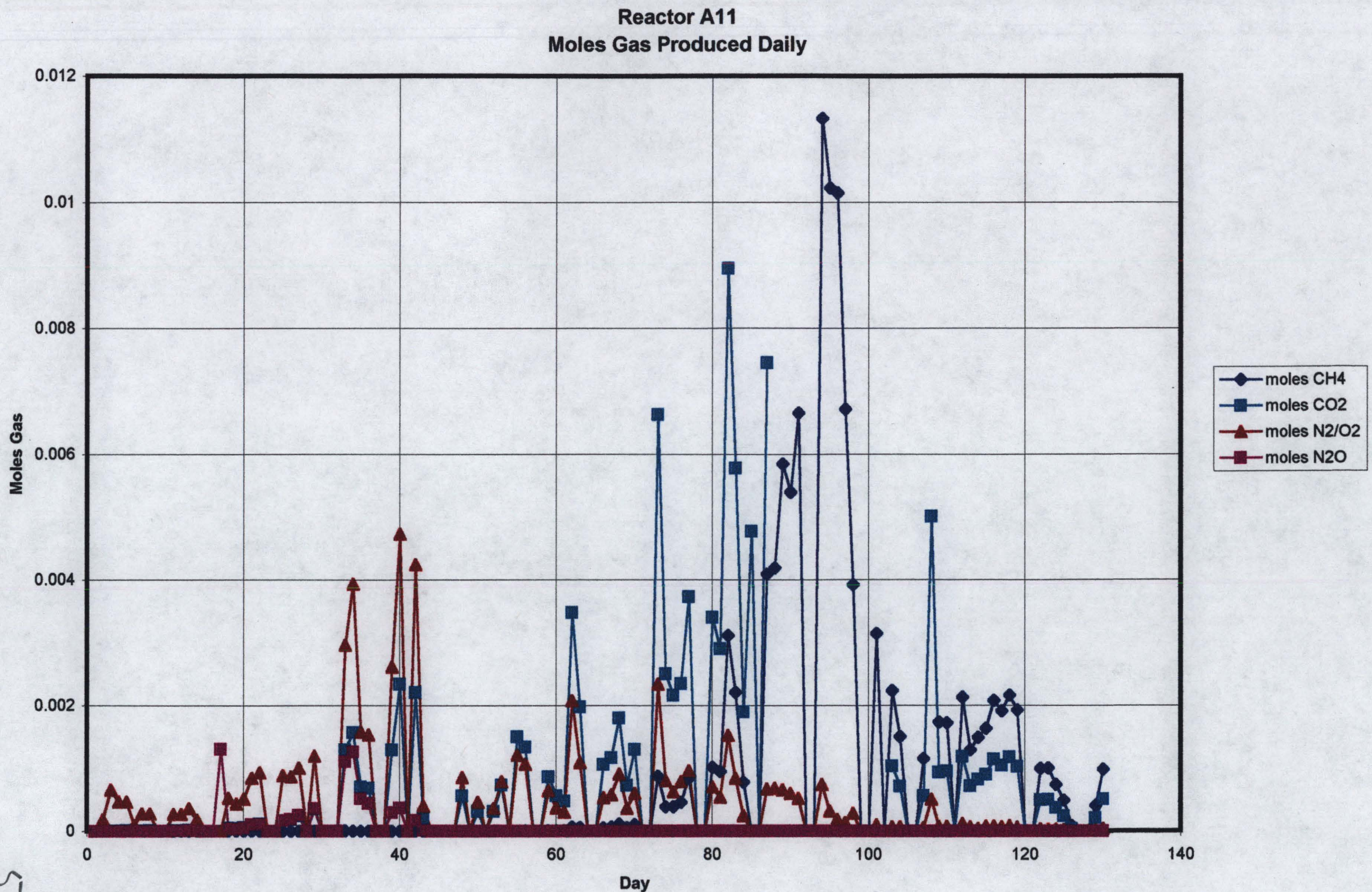
Reactor A8
Moles Gas Produced Daily
0.06
0.05
0.04
0.03
0.02
0.01
0
-0.01
Moles
0
5
10
15
20
25
30
35
40
45
50
Day
moles CH4
moles CO2
moles N2/O2
moles N2O

57



Reactor A10
Moles Gas Produced Daily
0.0002
0.0001
0
-0.0001
-0.0002
-0.0003
-0.0004
Moles
0
5
10
15
20
25
30
35
40
45
50
Day
moles CH4
moles CO2
moles N2/O2
moles N2O

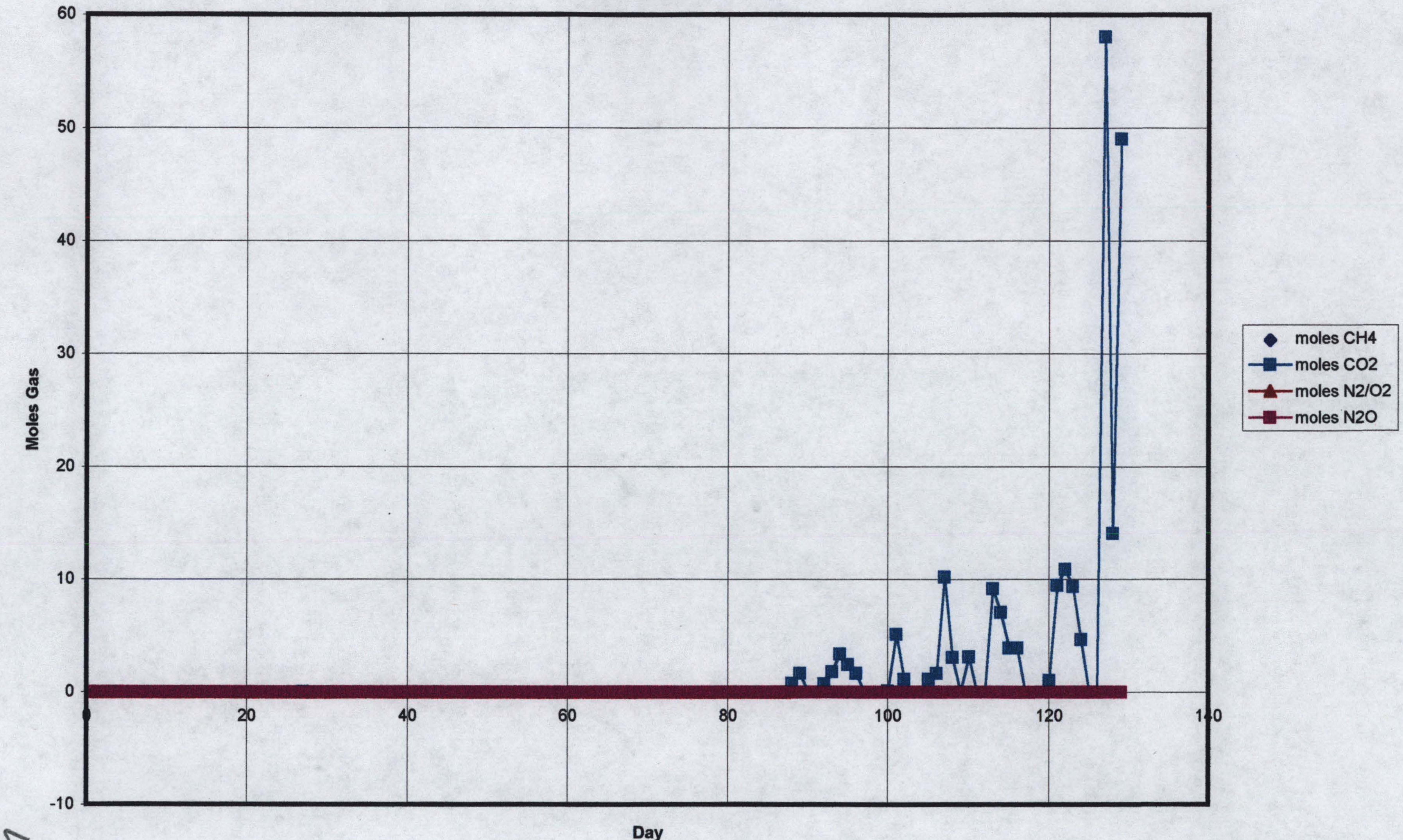
55



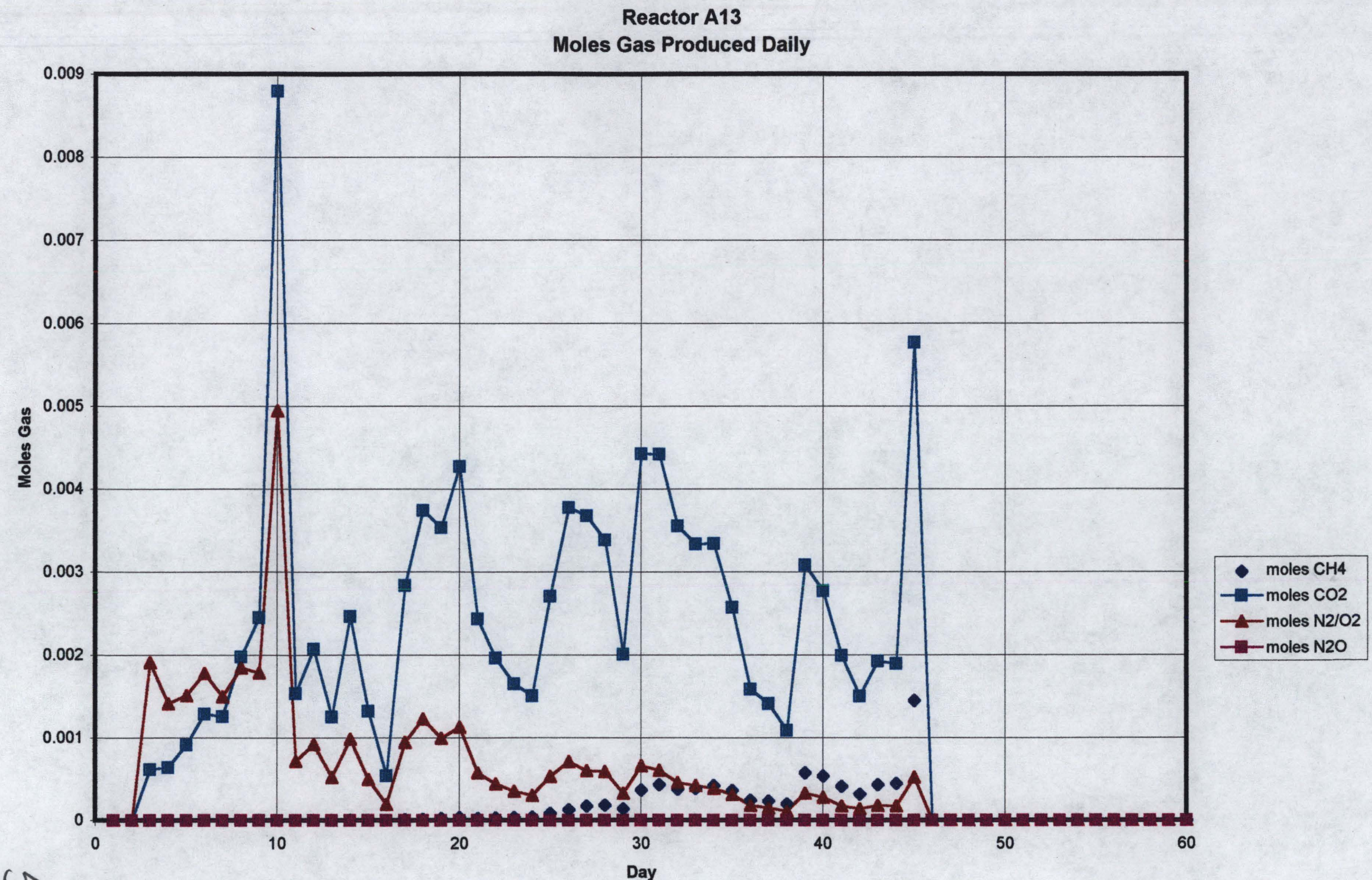
Reactor A11
Moles Gas Produced Daily
Moles Gas
0.012
0.01
0.008
0.006
0.004
0.002
0
0
20
40
60
80
100
120
140
Day
moles CH4
moles CO2
moles N2/O2
moles N2O

56

Reactor A12
Moles Gas Produced Daily

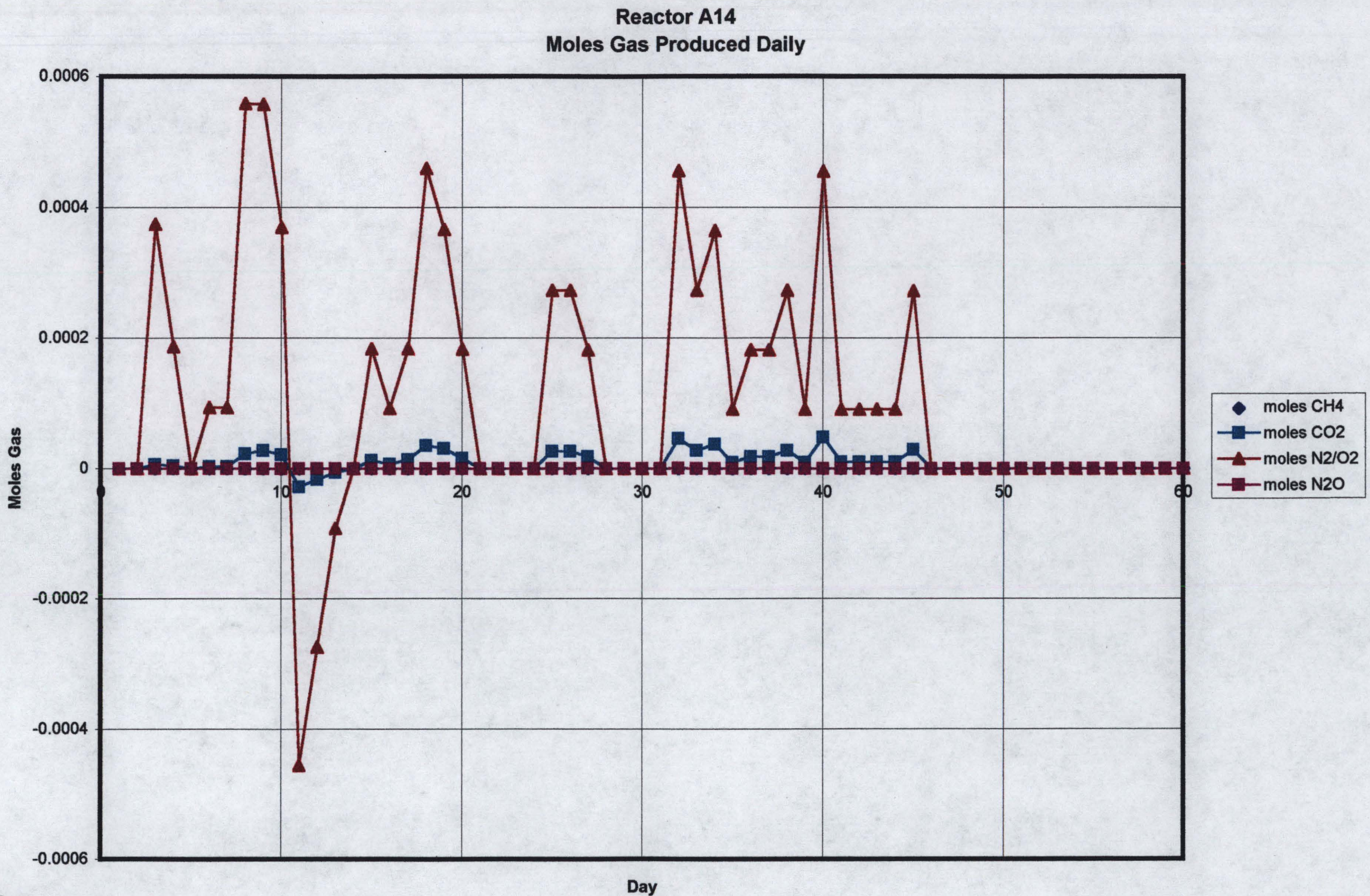


5

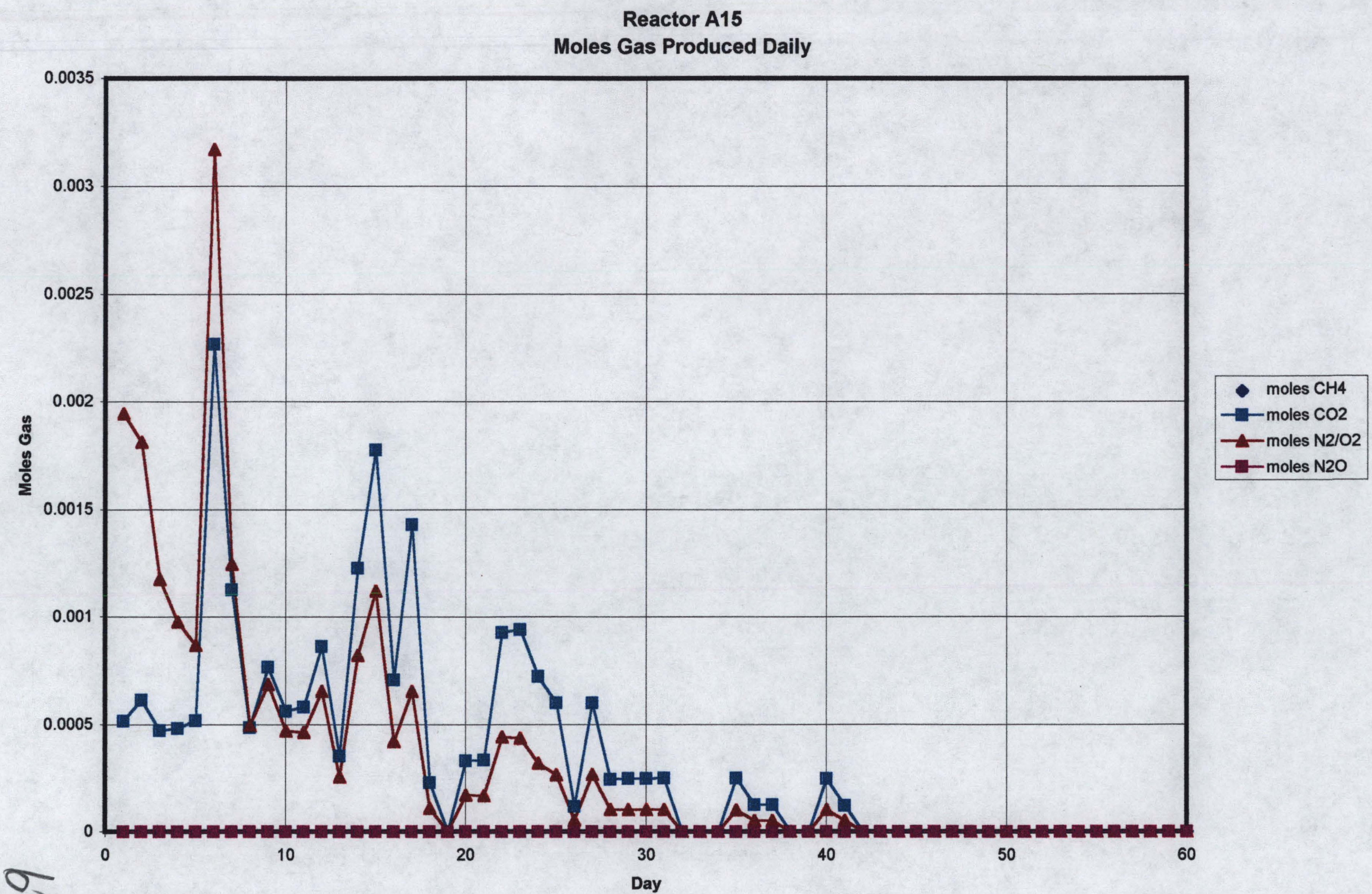


Reactor A13
Moles Gas Produced Daily
Moles Gas
0.009
0.008
0.007
0.006
0.005
0.004
0.003
0.002
0.001
0
0
10
20
30
40
50
60
Day
moles CH4
moles CO2
moles N2/O2
moles N2O

85


Reactor A14
Moles Gas Produced Daily
0.0006
0.0004
0.0002
0
-0.0002
-0.0004
-0.0006
Moles Gas
0
10
20
30
40
50
60
Day
moles CH4
moles CO2
moles N2/O2
moles N2O

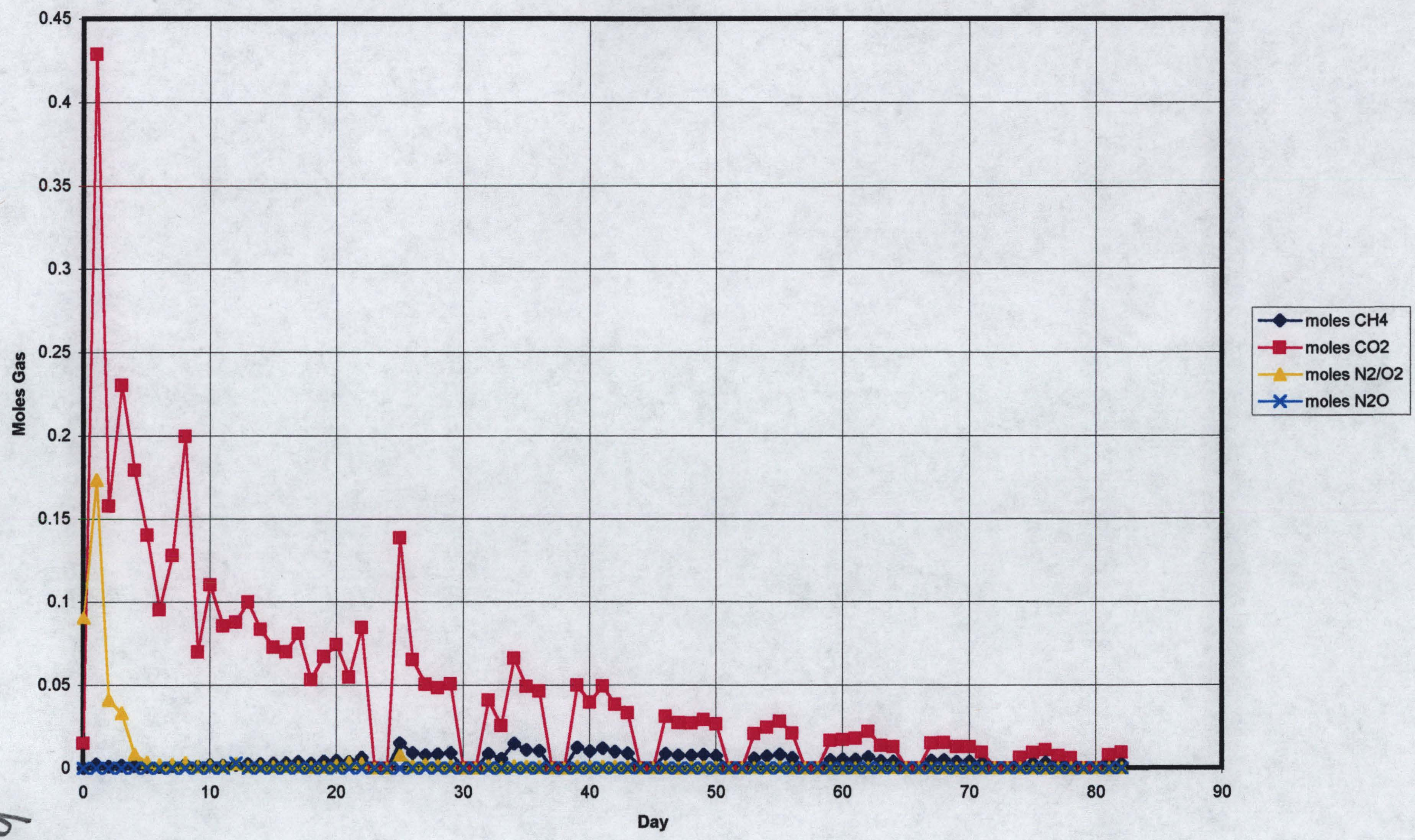
64



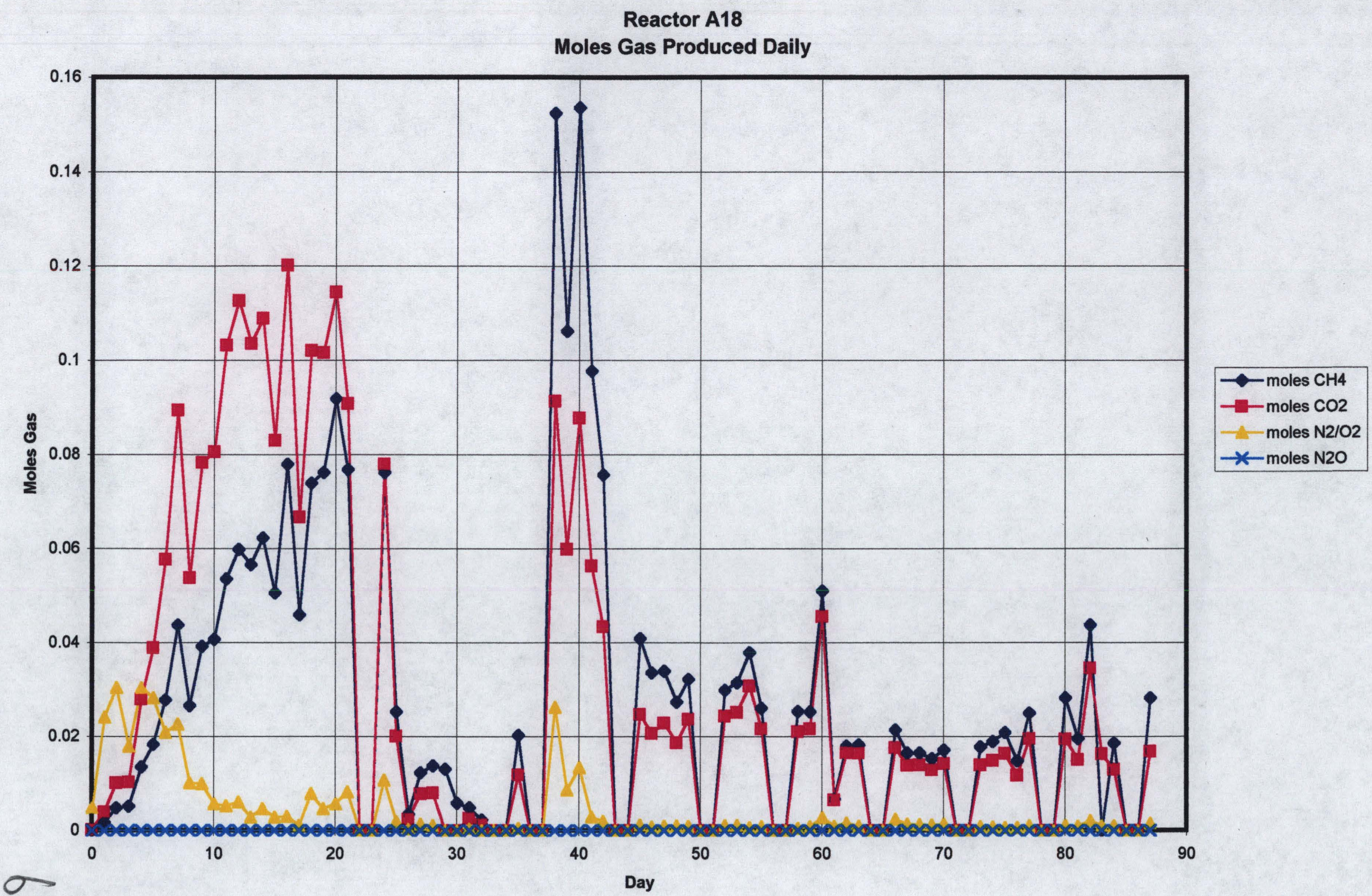
Reactor A15
Moles Gas Produced Daily
Moles Gas
0.0035
0.003
0.0025
0.002
0.0015
0.001
0.0005
0
0
10
20
30
40
50
60
Day
moles CH4
moles CO2
moles N2/O2
moles N2O

60

Reactor A16
Moles Gas Produced Daily

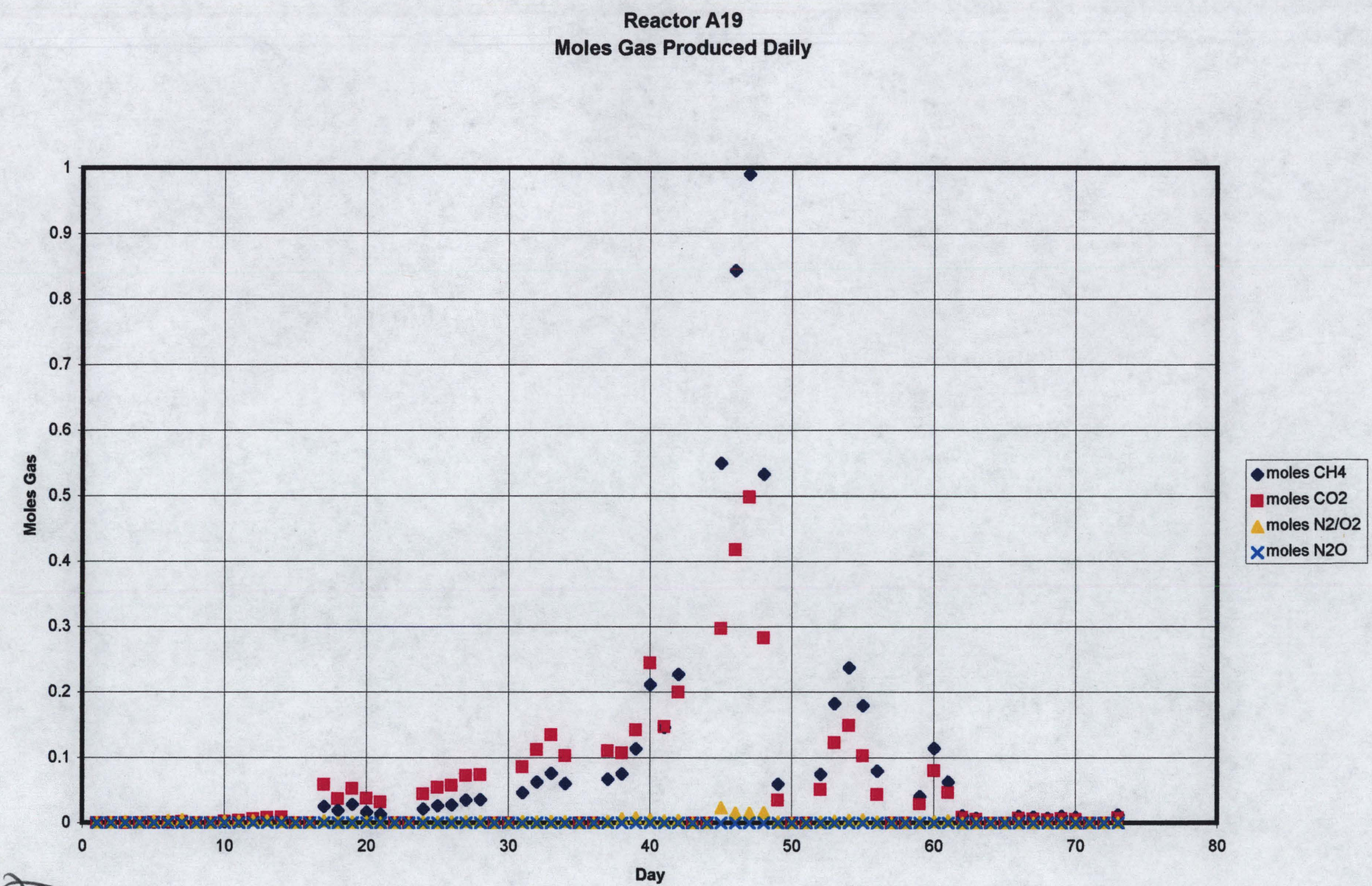


19

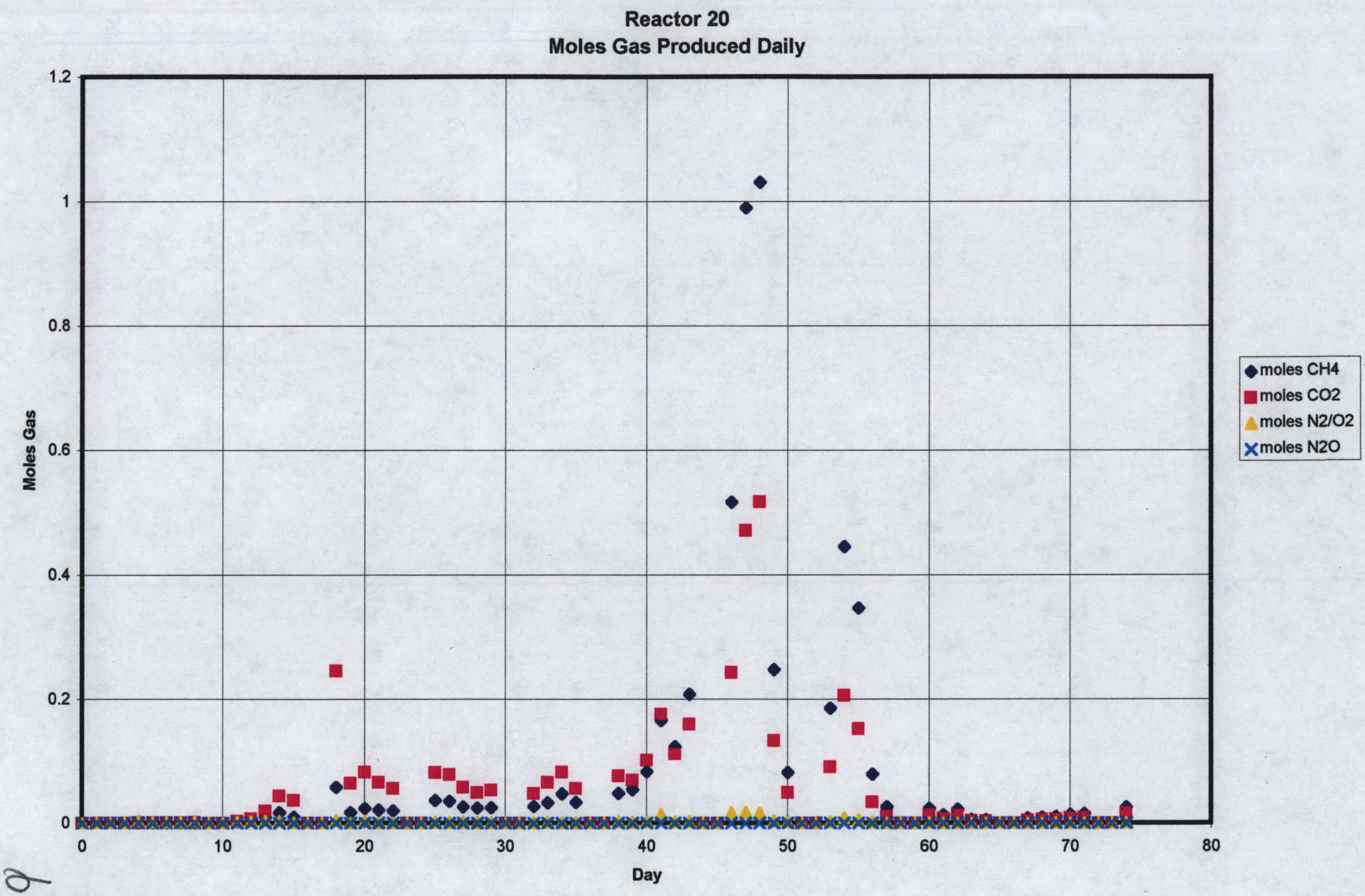


Reactor A18
Moles Gas Produced Daily
Moles Gas
Day
0.16
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
0
10
20
30
40
50
60
70
80
90
moles CH4
moles CO2
moles N2/O2
moles N2O

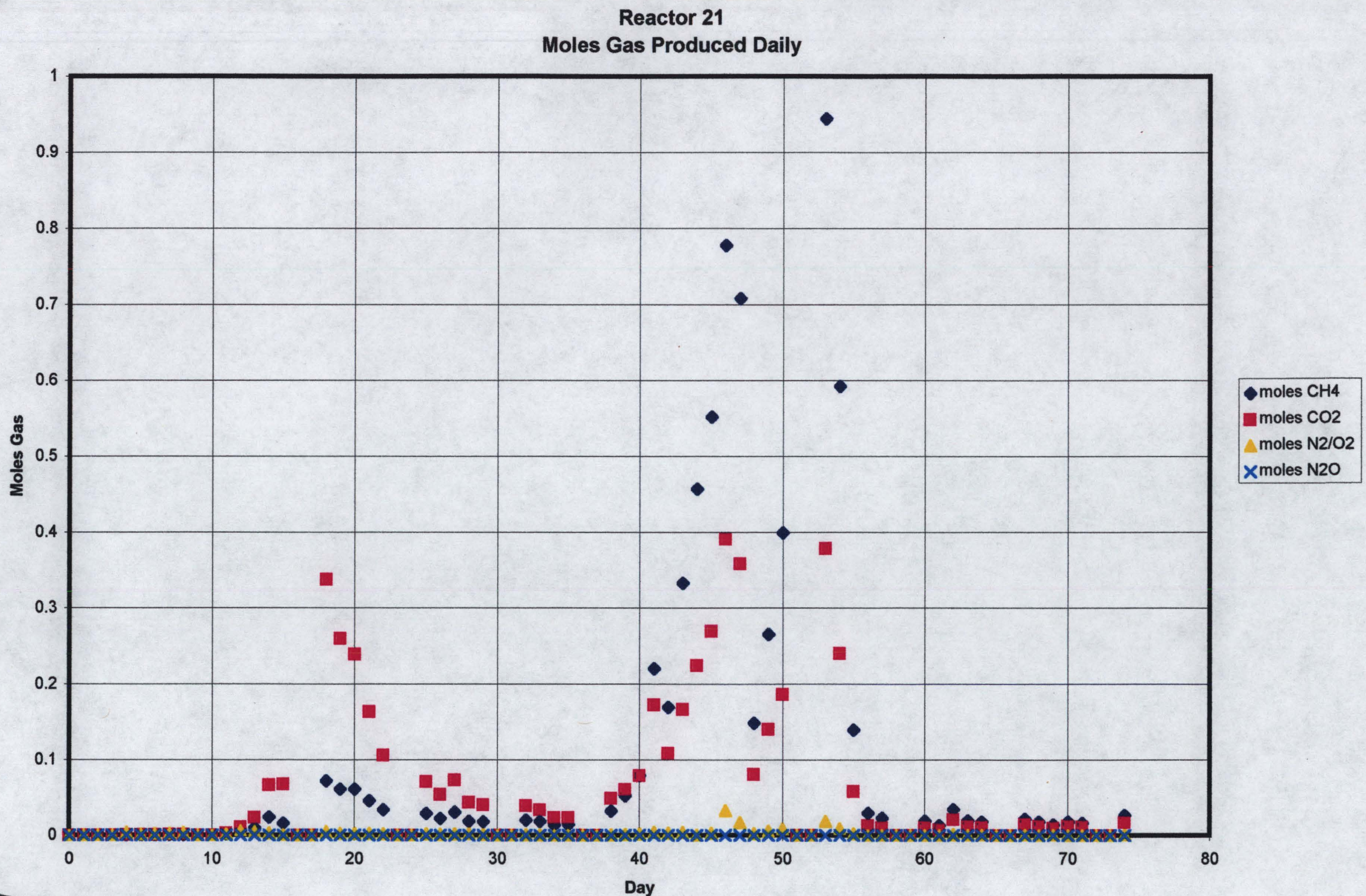
62


Reactor A19
Moles Gas Produced Daily
Moles Gas
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
10
20
30
40
50
60
70
80
Day
moles CH4
moles CO2
moles N2/O2
moles N2O

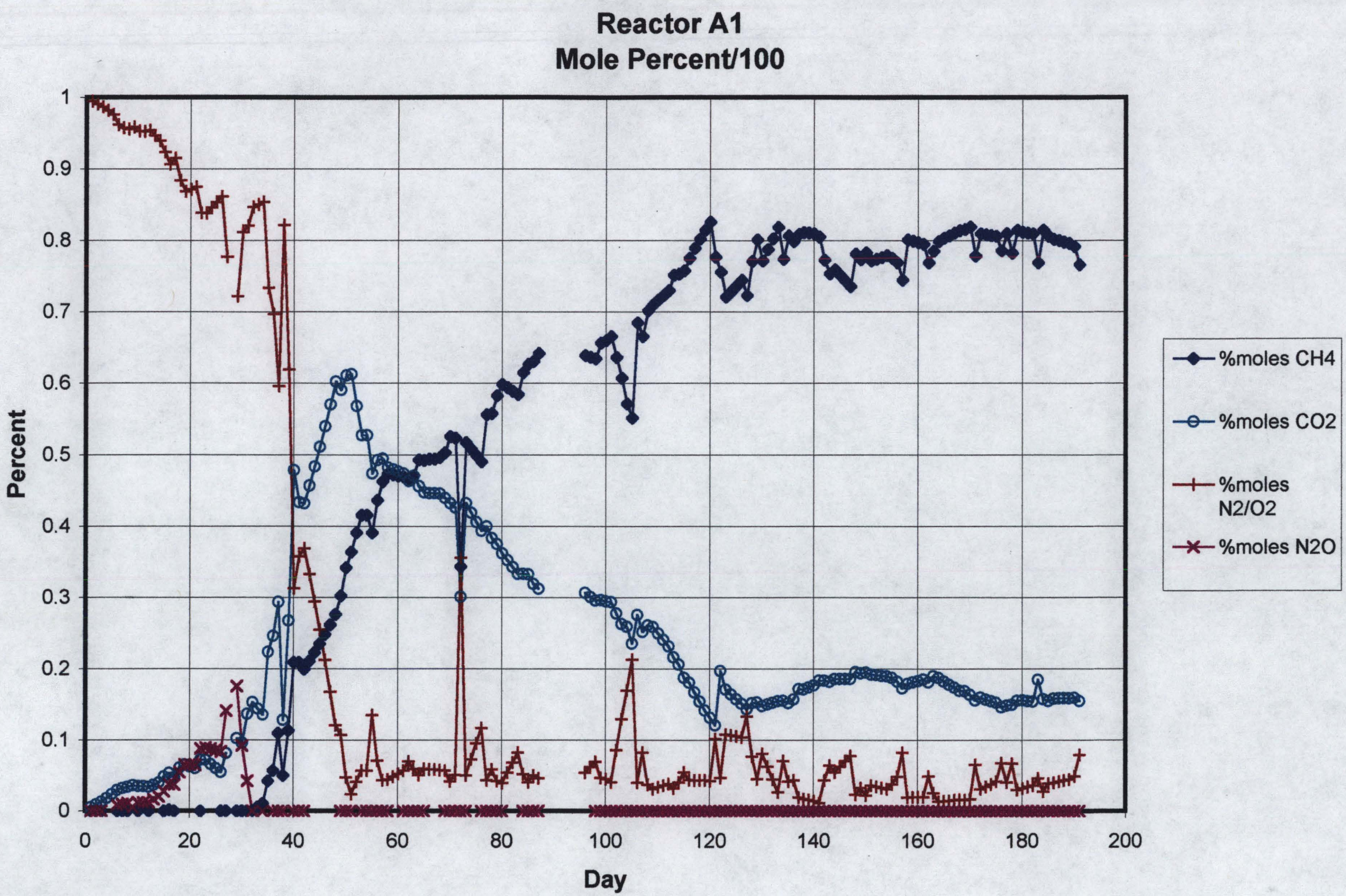
63


Reactor 20
Moles Gas Produced Daily
Moles Gas
1.2
1
0.8
0.6
0.4
0.2
0
0
10
20
30
40
50
60
70
80
Day
moles CH4
moles CO2
moles N2/O2
moles N2O

69

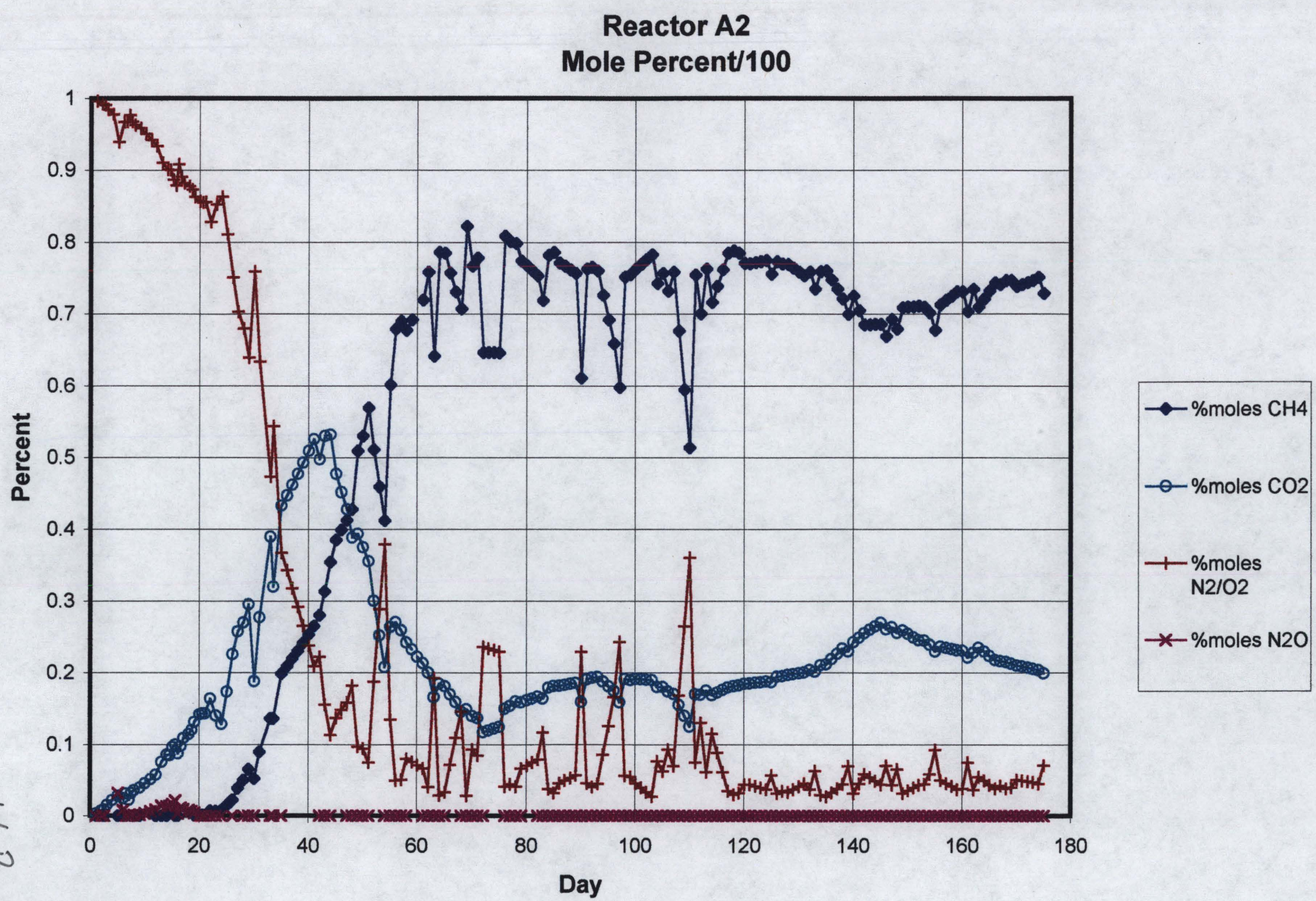

Reactor 21
Moles Gas Produced Daily
Moles Gas
Day
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
10
20
30
40
50
60
70
80
moles CH4
moles CO2
moles N2/O2
moles N2O

59



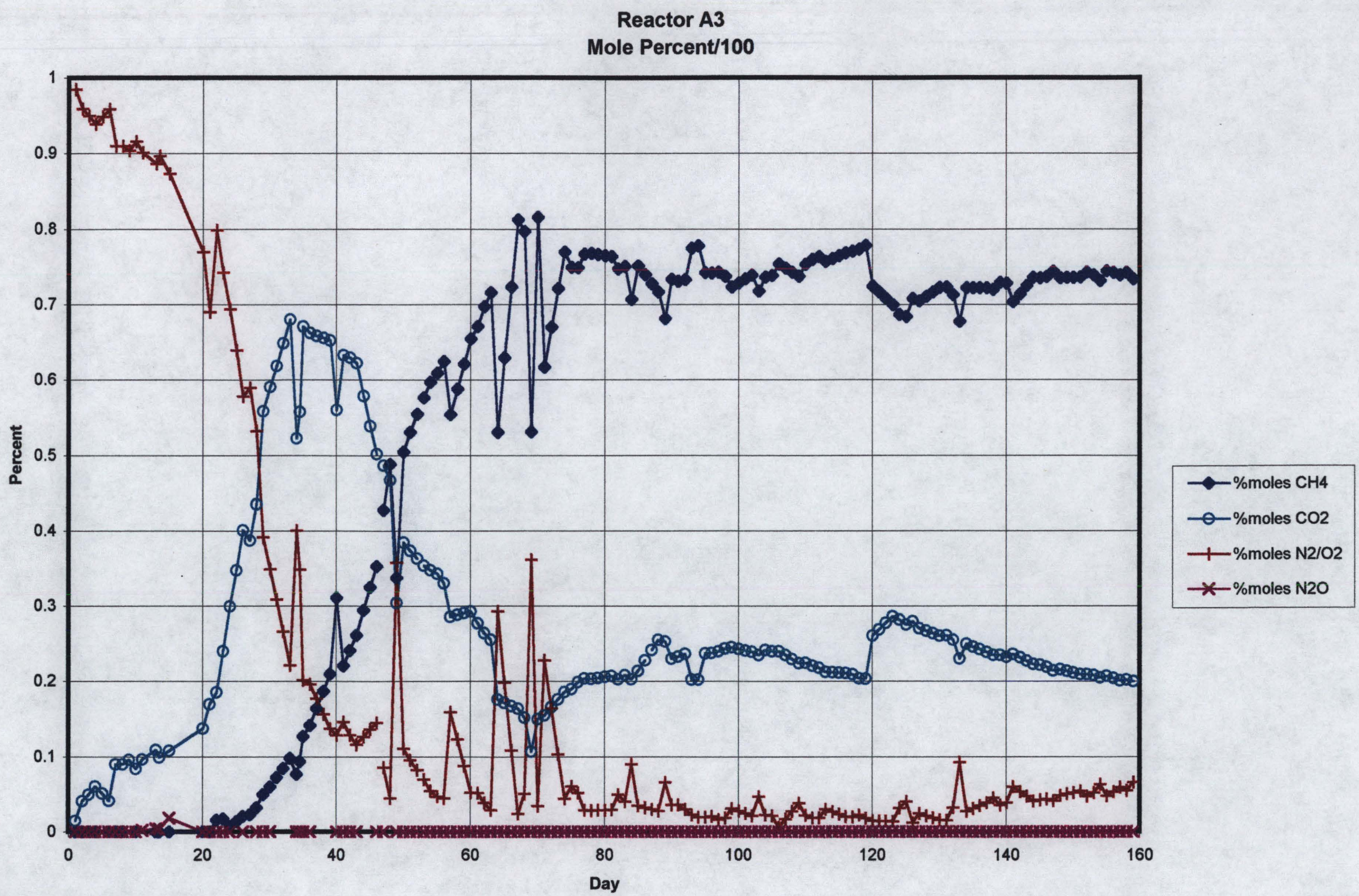
Reactor A1
Mole Percent/100
Percent
Day
%moles CH4
%moles CO2
%moles N2/O2
%moles N2O
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1
0
20
40
60
80
100
120
140
160
180
200

664



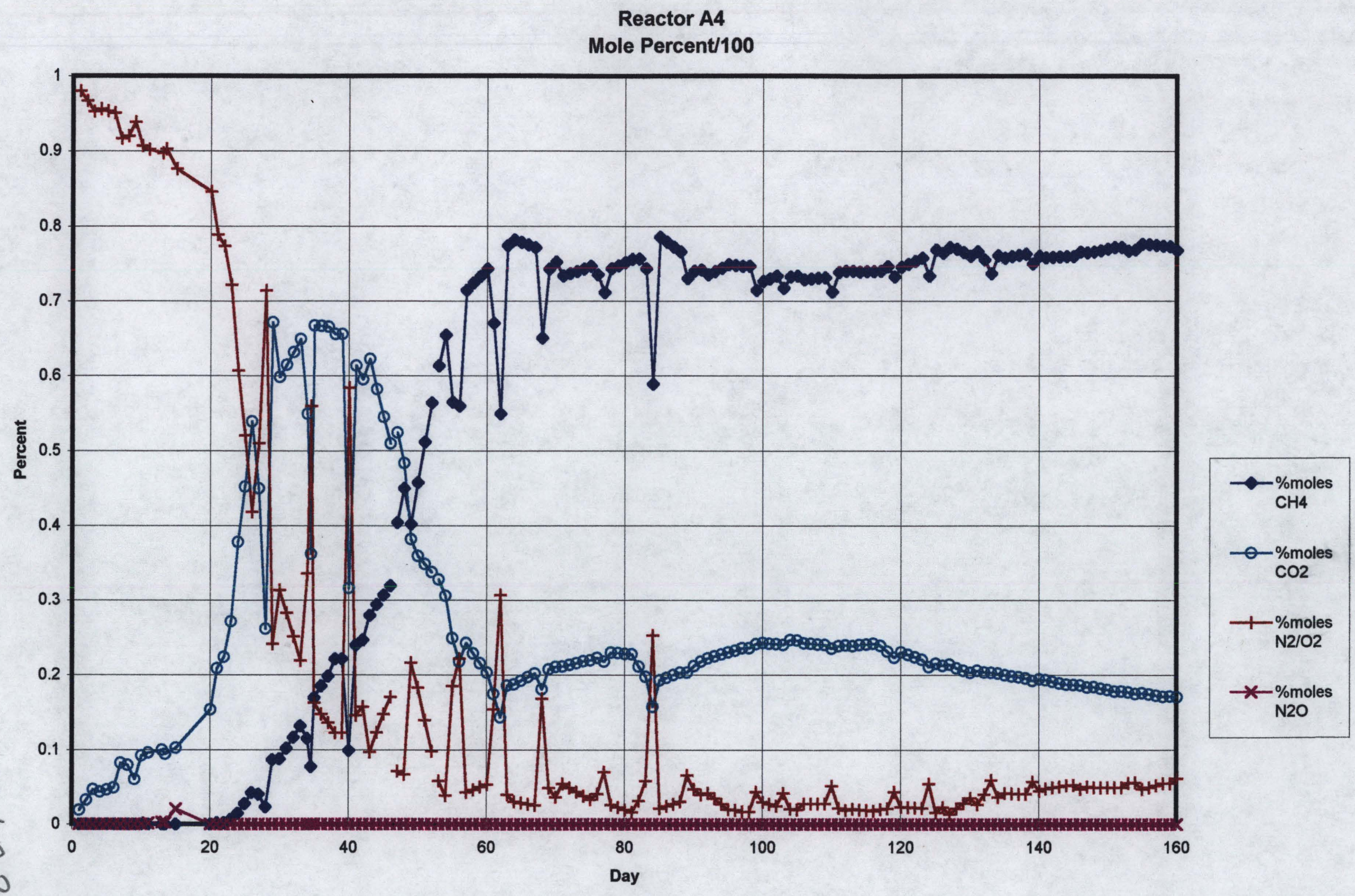
Reactor A2
Mole Percent/100
Percent
Day
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1
0
20
40
60
80
100
120
140
160
180
%moles CH4
%moles CO2
%moles N2/O2
%moles N2O

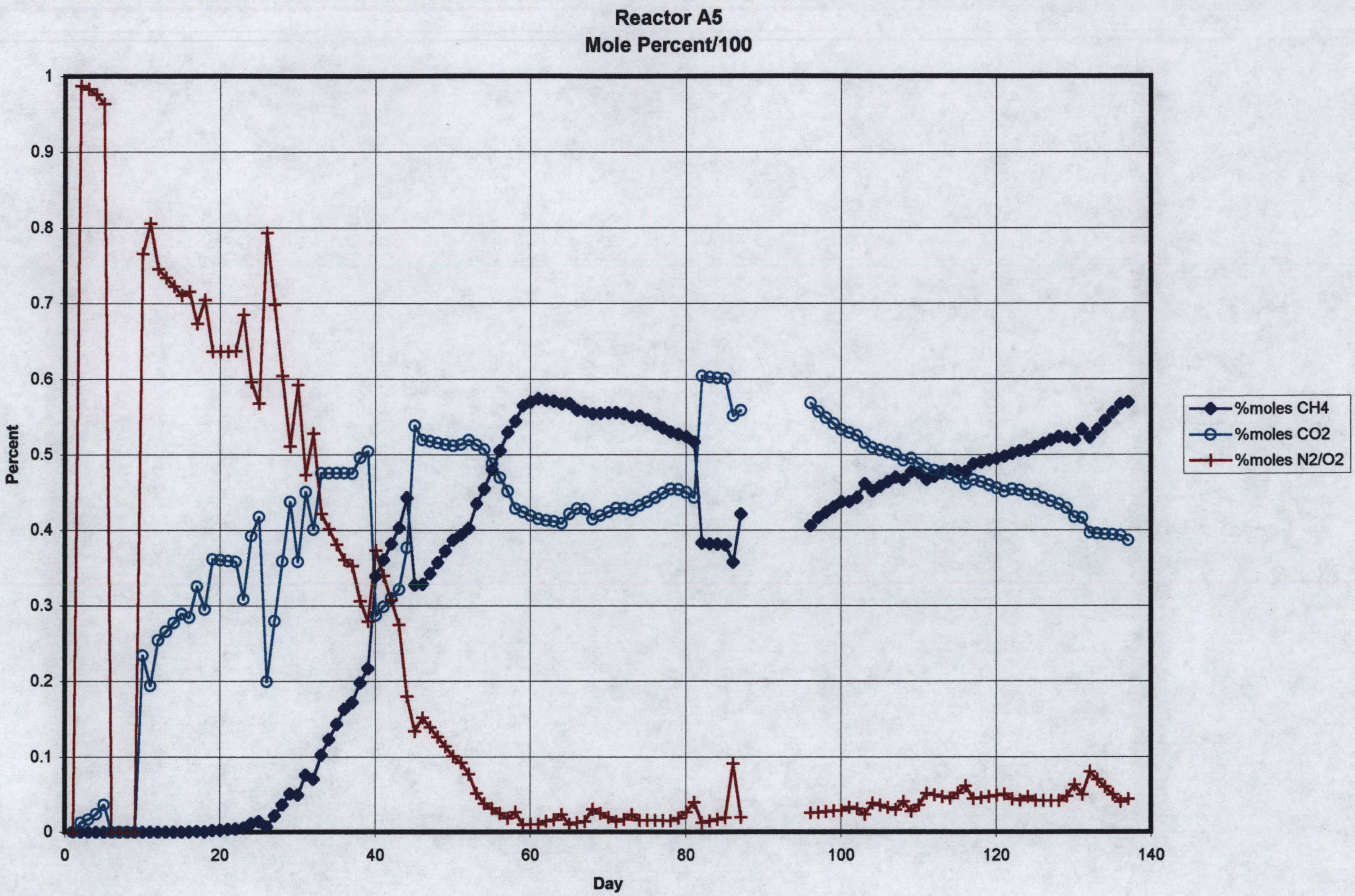
Q99



Reactor A3
Mole Percent/100
Percent
Day
%moles CH4
%moles CO2
%moles N2/O2
%moles N2O
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1
0
20
40
60
80
100
120
140
160

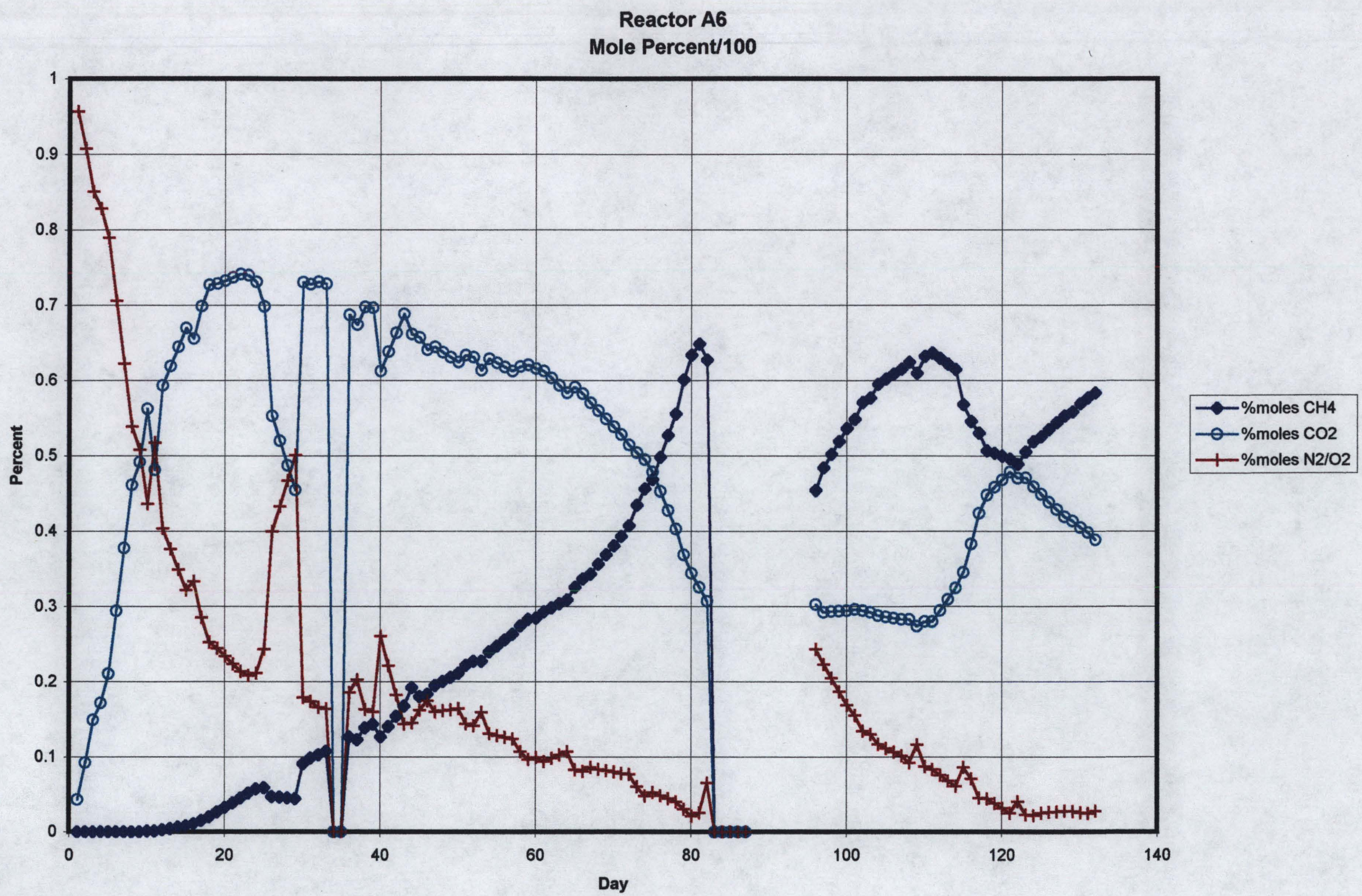
674


Reactor A4
Mole Percent/100
Percent
Day
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1
0
20
40
60
80
100
120
140
160
%moles CH4
%moles CO2
%moles N2/O2
%moles N2O



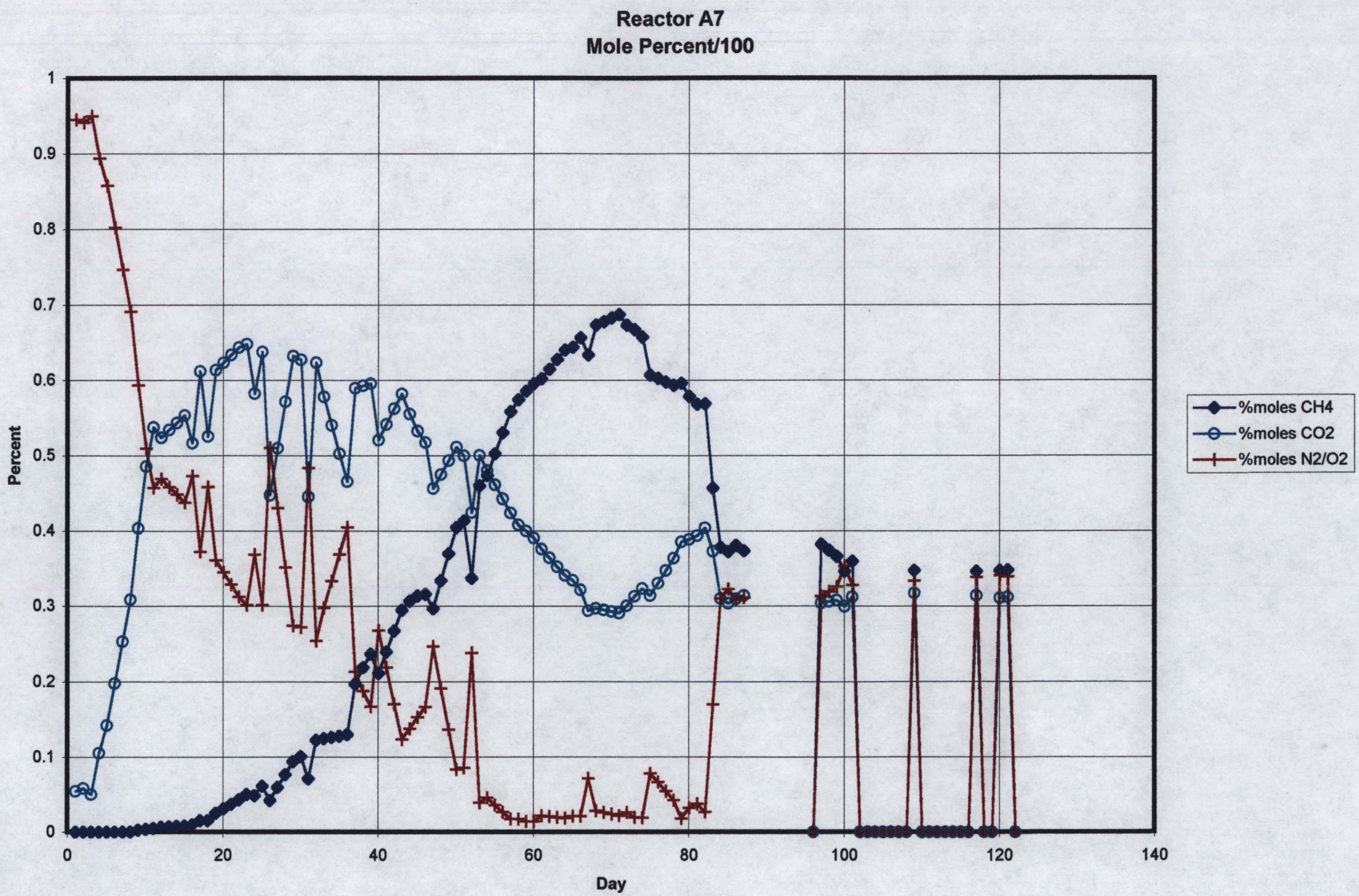
Reactor A5
Mole Percent/100
Percent
Day
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
20
40
60
80
100
120
140
%moles CH4
%moles CO2
%moles N2/O2

687

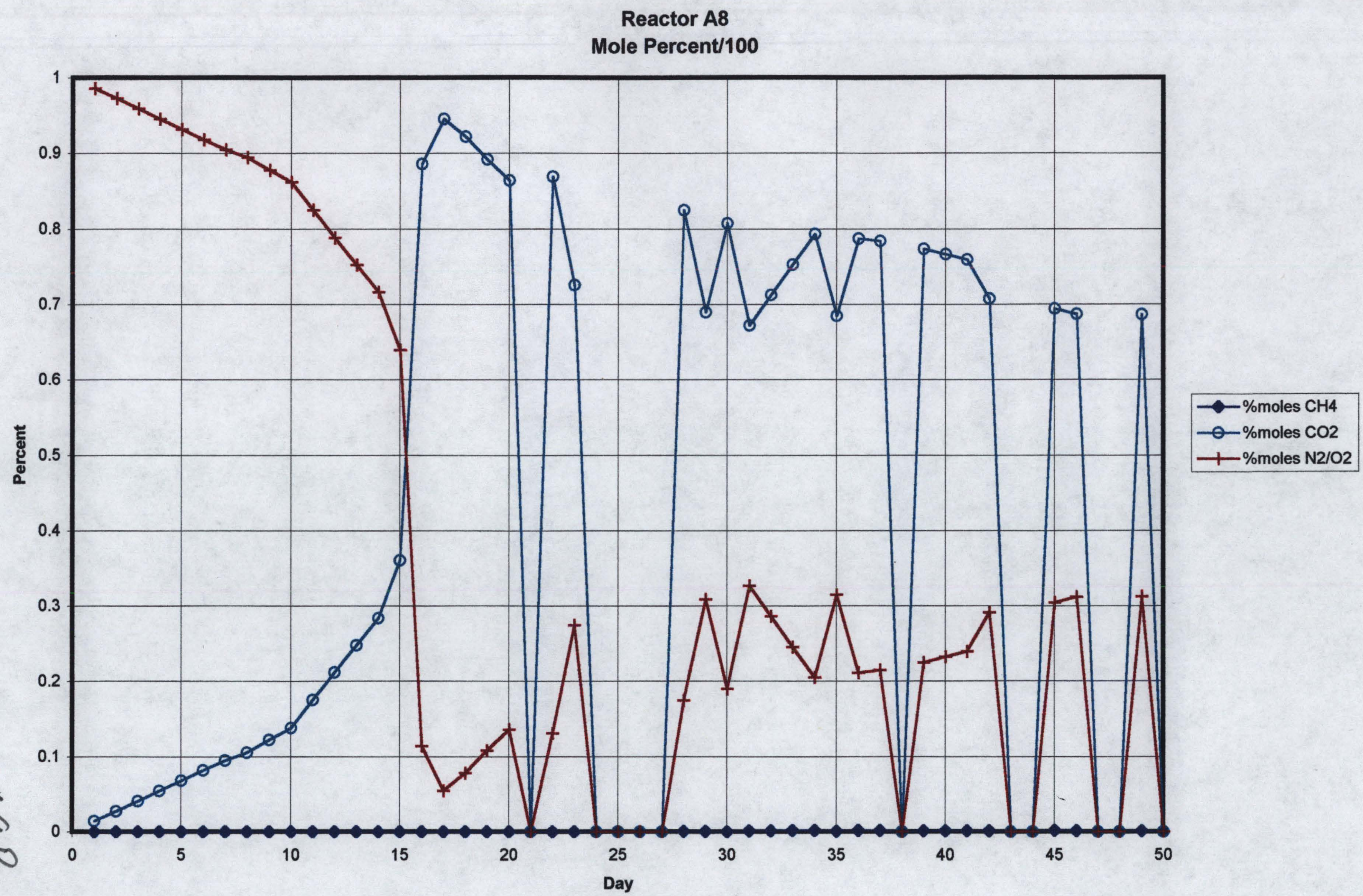


Reactor A6
Mole Percent/100
Percent
Day
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1
0
20
40
60
80
100
120
140
%moles CH4
%moles CO2
%moles N2/O2

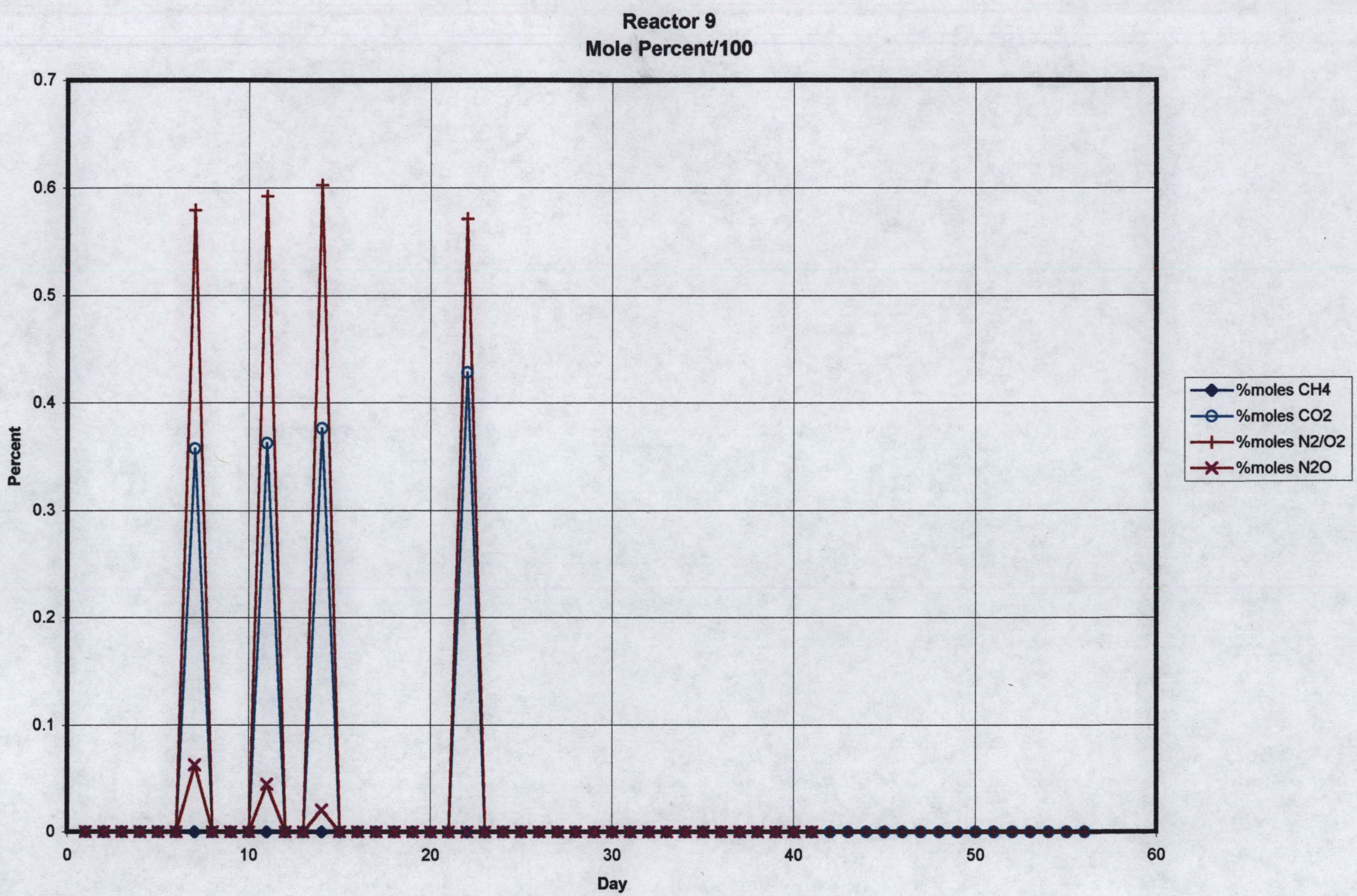
689


Reactor A7
Mole Percent/100
Percent
Day
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1
0
20
40
60
80
100
120
140
%moles CH4
%moles CO2
%moles N2/O2

69A

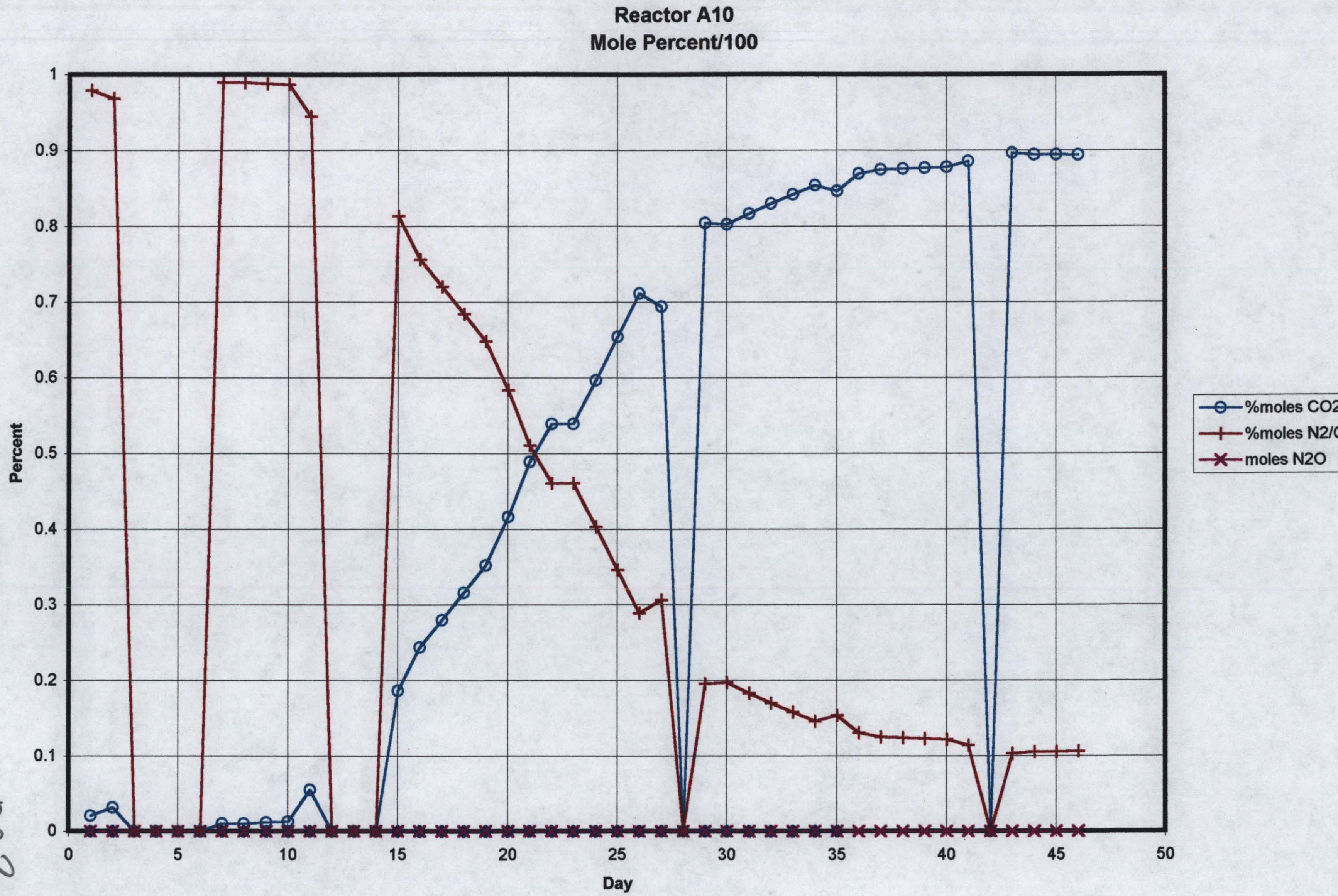

Reactor A8
Mole Percent/100
Percent
Day
%moles CH4
%moles CO2
%moles N2/O2
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1
0
5
10
15
20
25
30
35
40
45
50

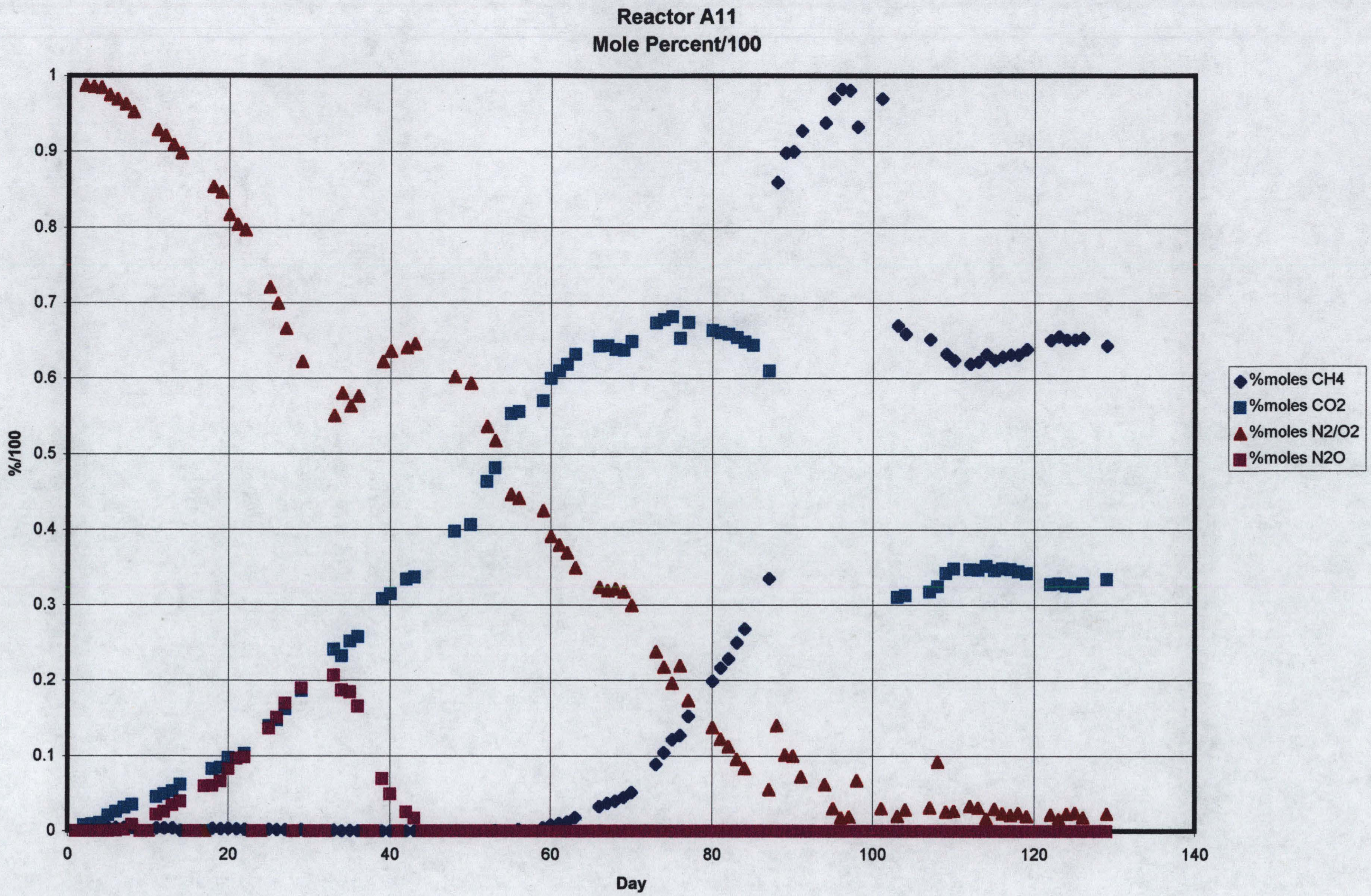
69B



Reactor 9
Mole Percent/100
Percent
Day
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0
10
20
30
40
50
60
%moles CH4
%moles CO2
%moles N2/O2
%moles N2O

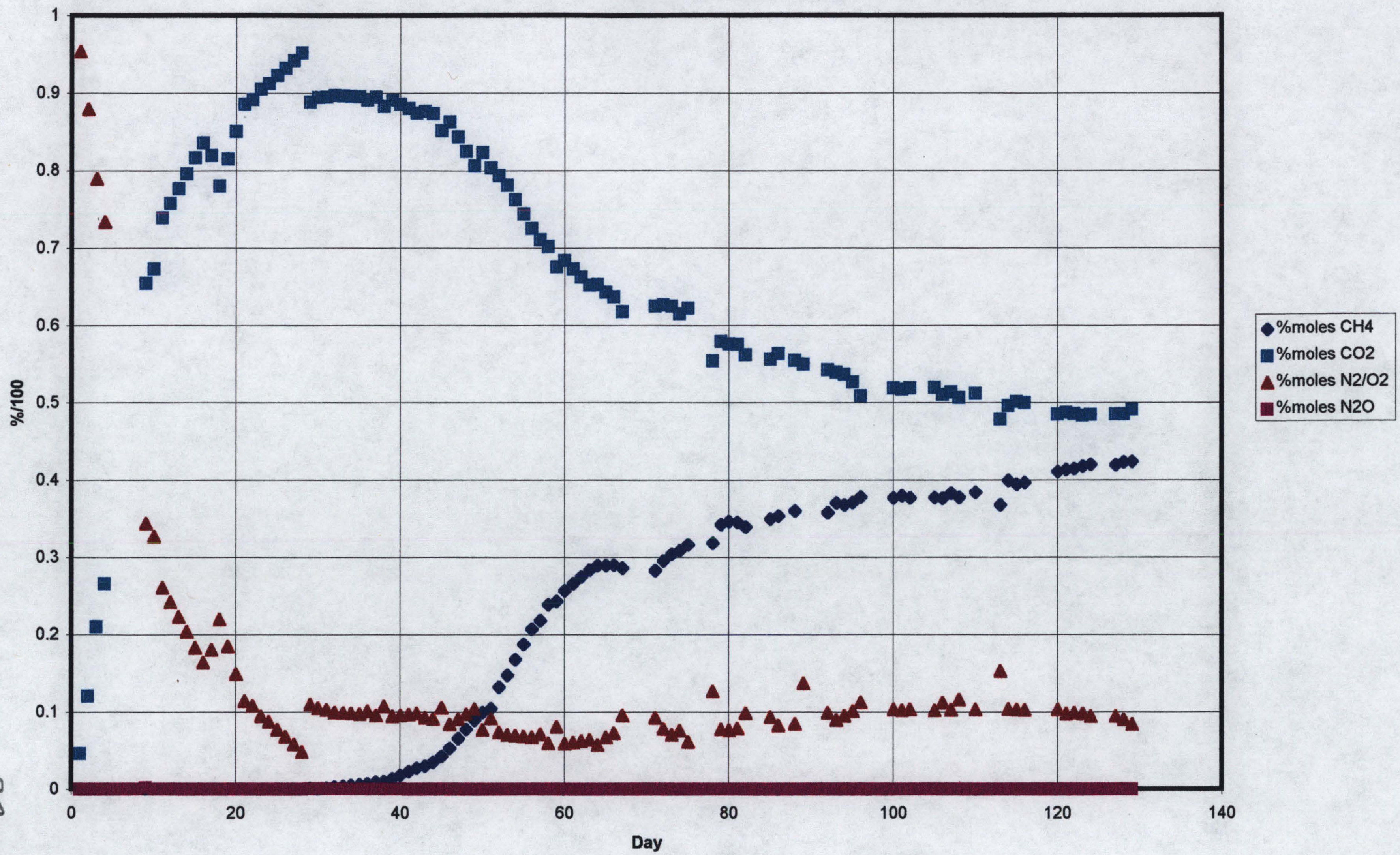
1047


Reactor A10
Mole Percent/100
Percent
Day
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1
0
5
10
15
20
25
30
35
40
45
50
%moles CO2
%moles N2/O2
moles N2O

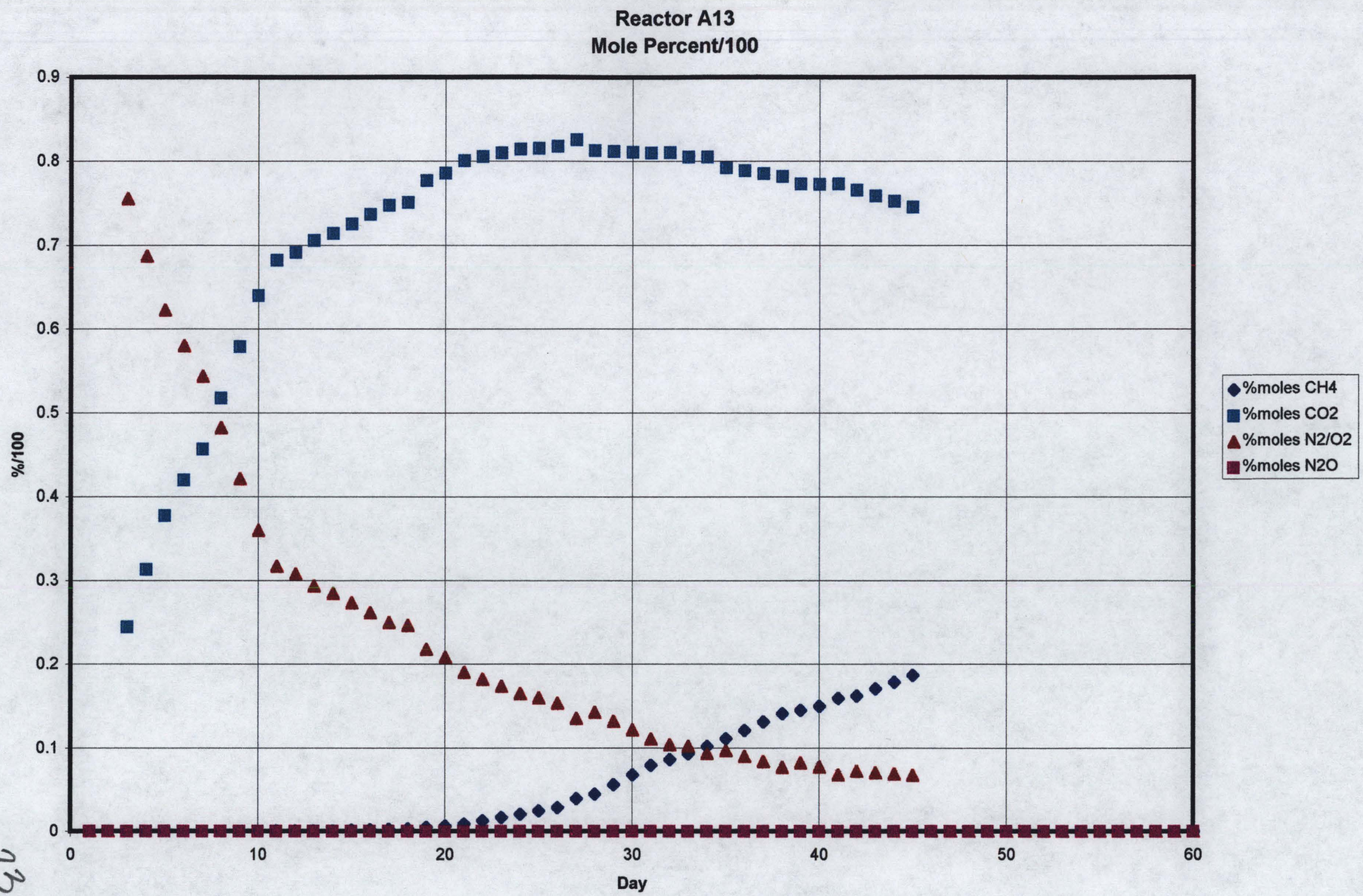

Reactor A11
Mole Percent/100
%/100
Day
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1
0
20
40
60
80
100
120
140
%moles CH4
%moles CO2
%moles N2/O2
%moles N2O

16

Reactor A12
Mole Percent/100

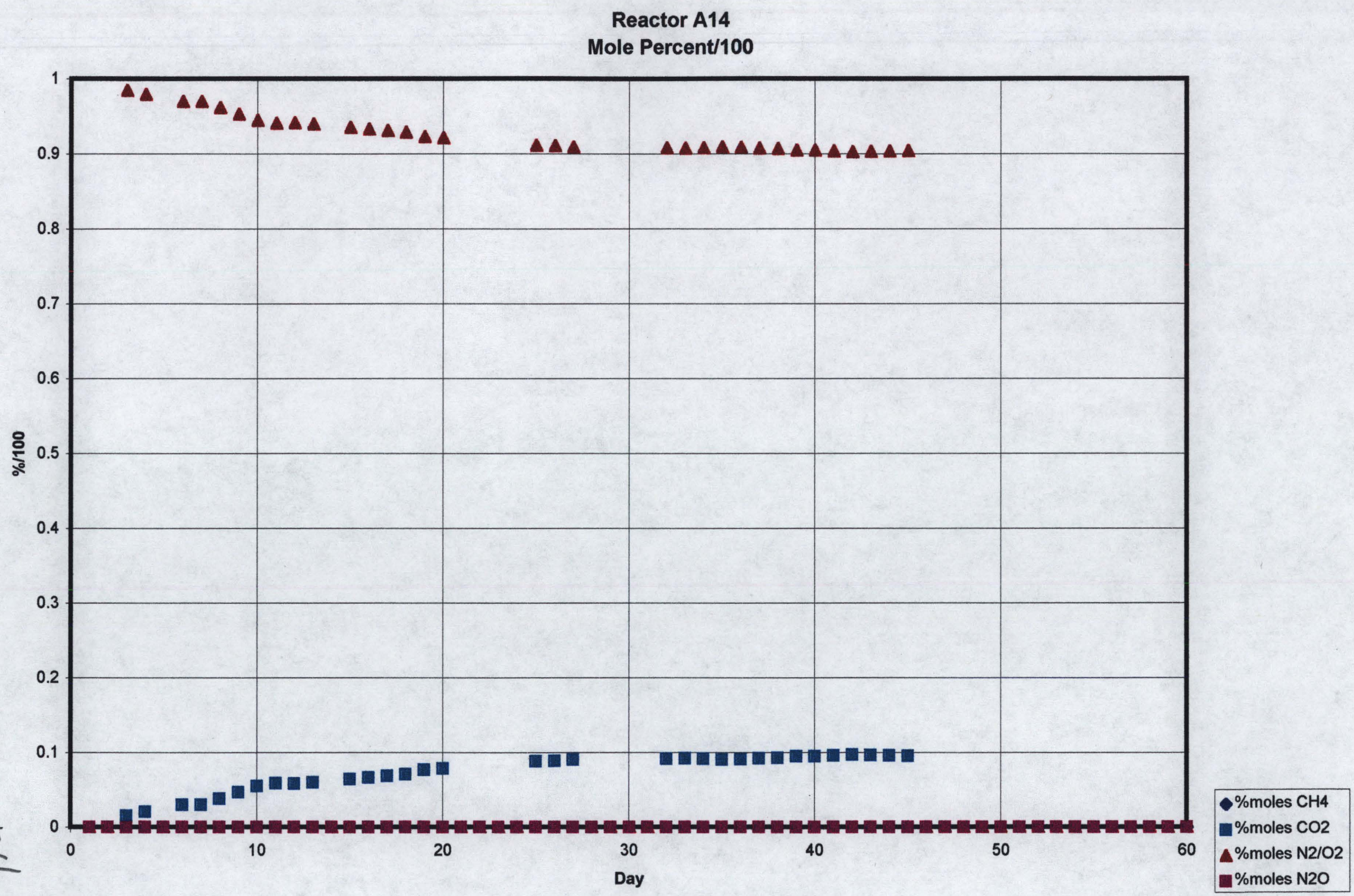


72



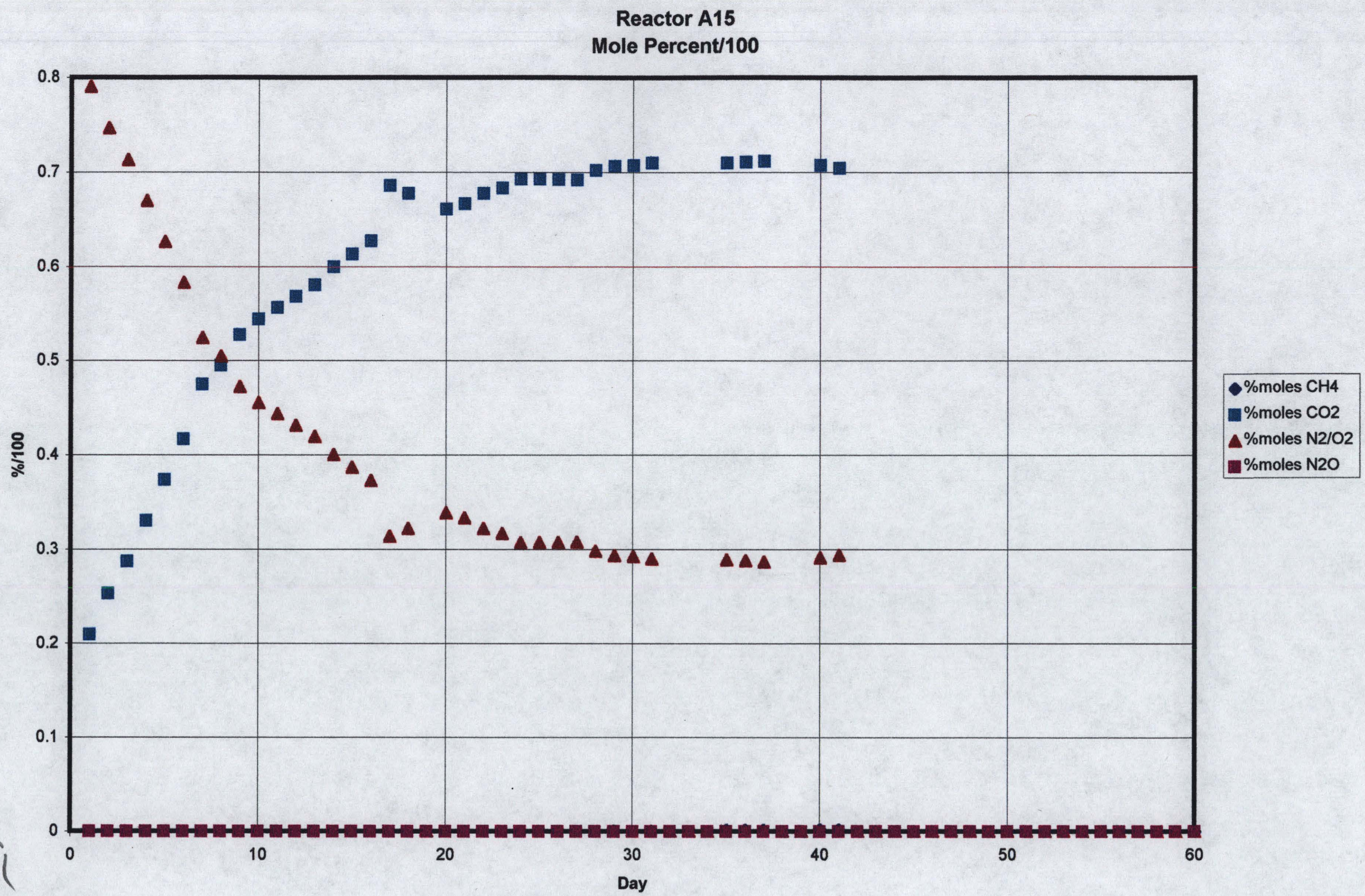
Reactor A13
Mole Percent/100
%/100
Day
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
10
20
30
40
50
60
%moles CH4
%moles CO2
%moles N2/O2
%moles N2O

63



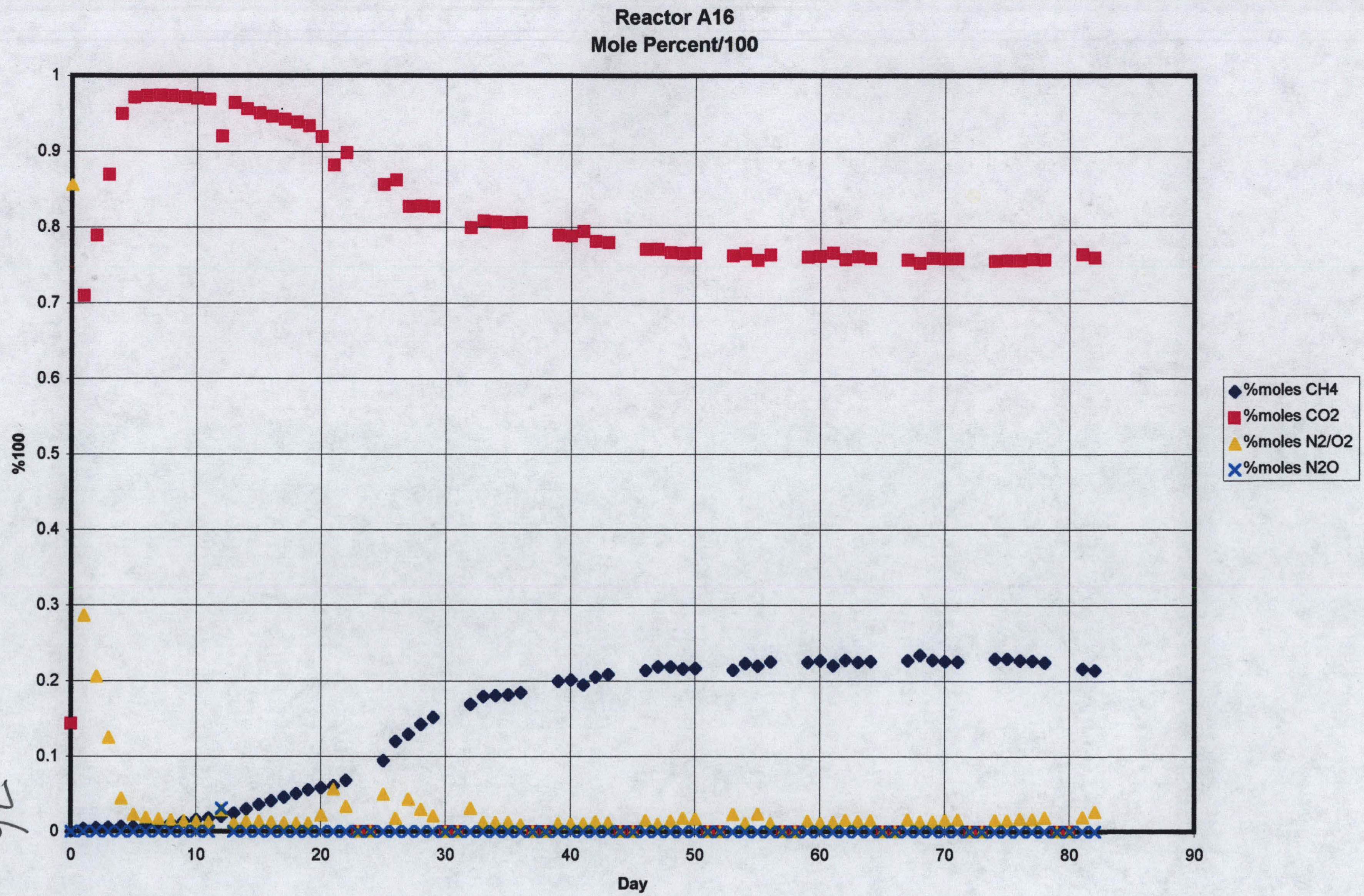
Reactor A14
Mole Percent/100
%/100
Day
%moles CH4
%moles CO2
%moles N2/O2
%moles N2O

84

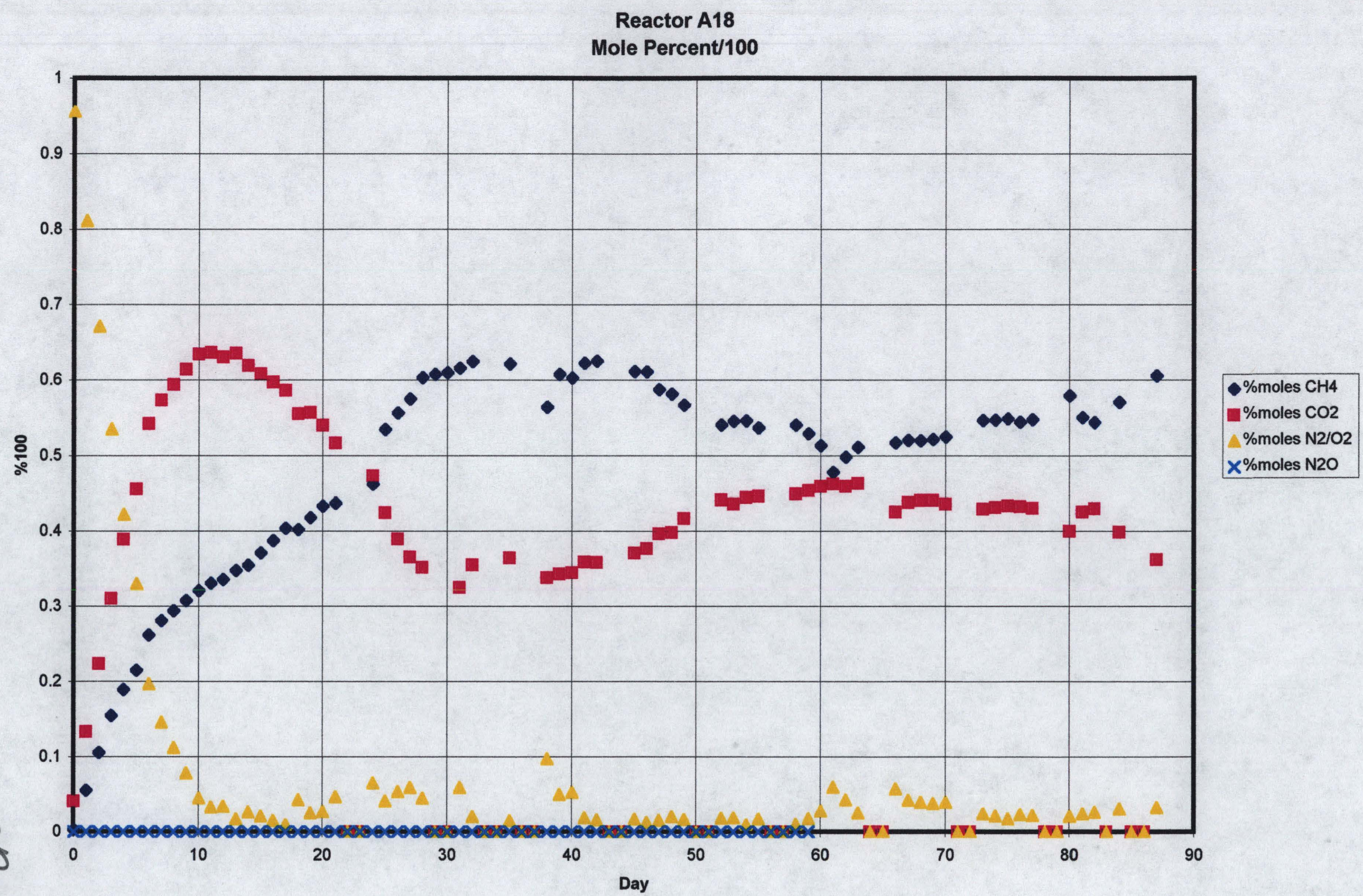


Reactor A15
Mole Percent/100
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
%/100
0
10
20
30
40
50
60
Day
%moles CH4
%moles CO2
%moles N2/O2
%moles N2O

75

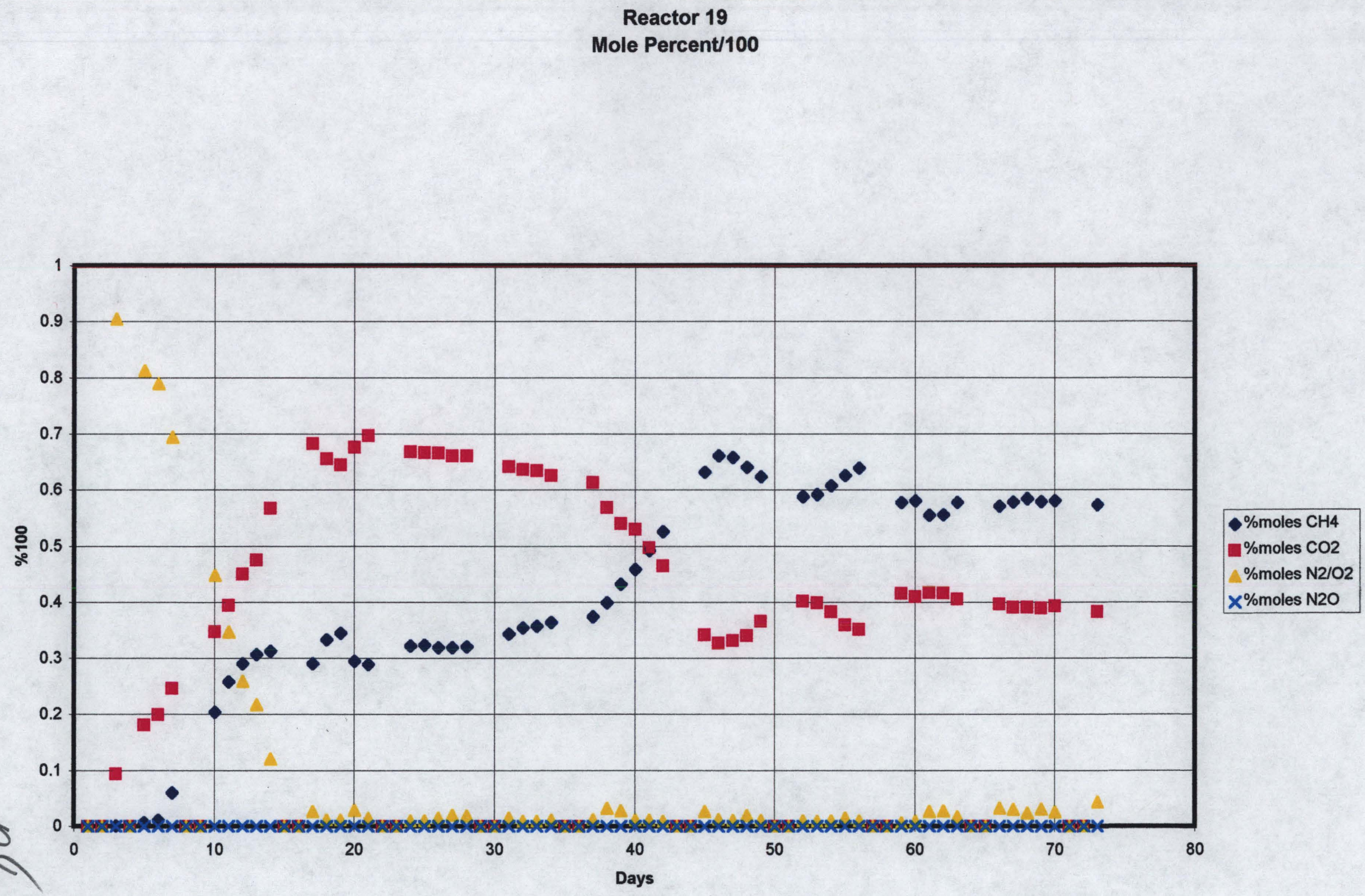

Reactor A16
Mole Percent/100
%100
Day
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1
0
10
20
30
40
50
60
70
80
90
%moles CH4
%moles CO2
%moles N2/O2
%moles N2O

96



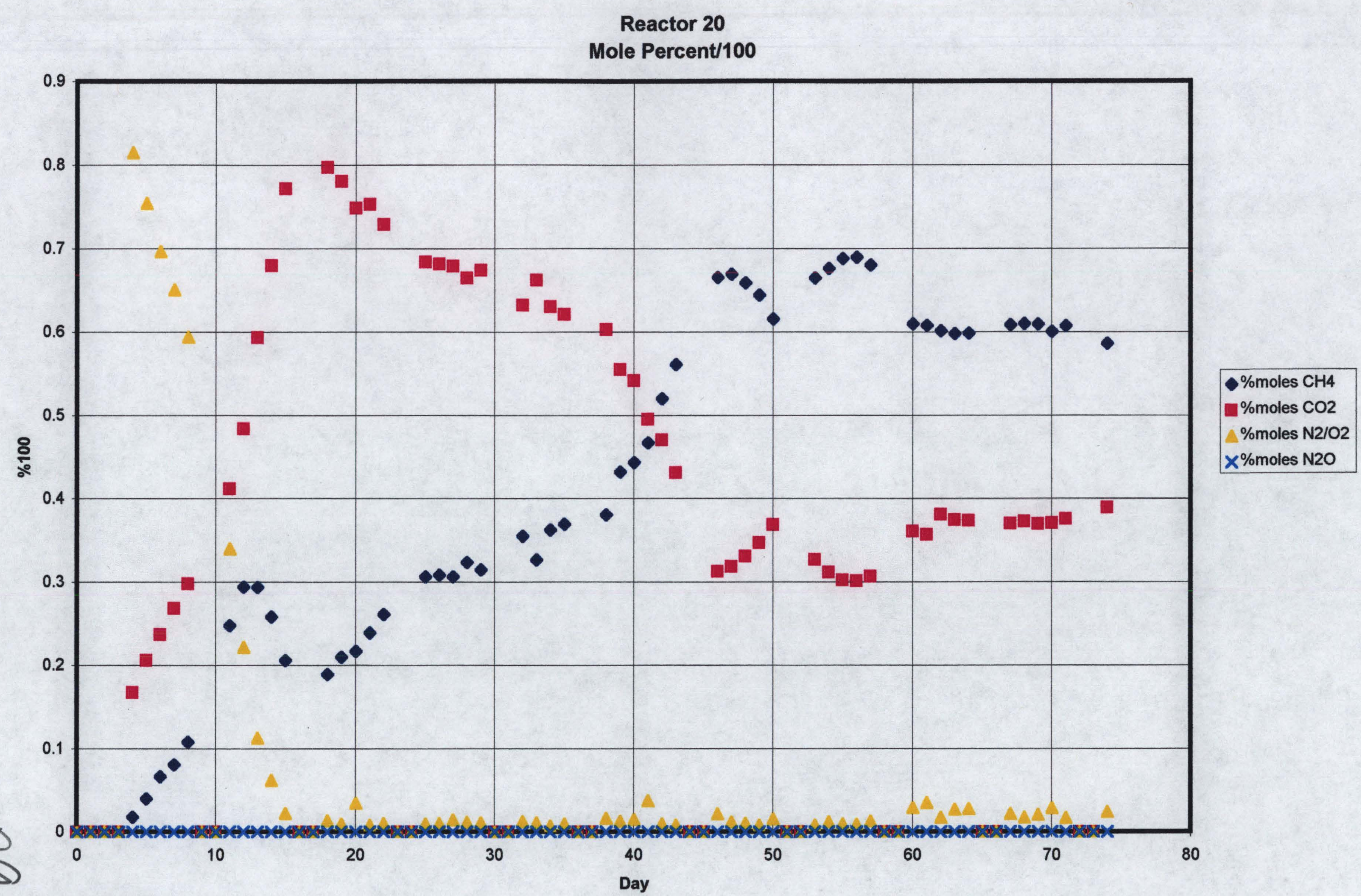
Reactor A18
Mole Percent/100
%100
Day
%moles CH4
%moles CO2
%moles N2/O2
%moles N2O
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1
0
10
20
30
40
50
60
70
80
90

44



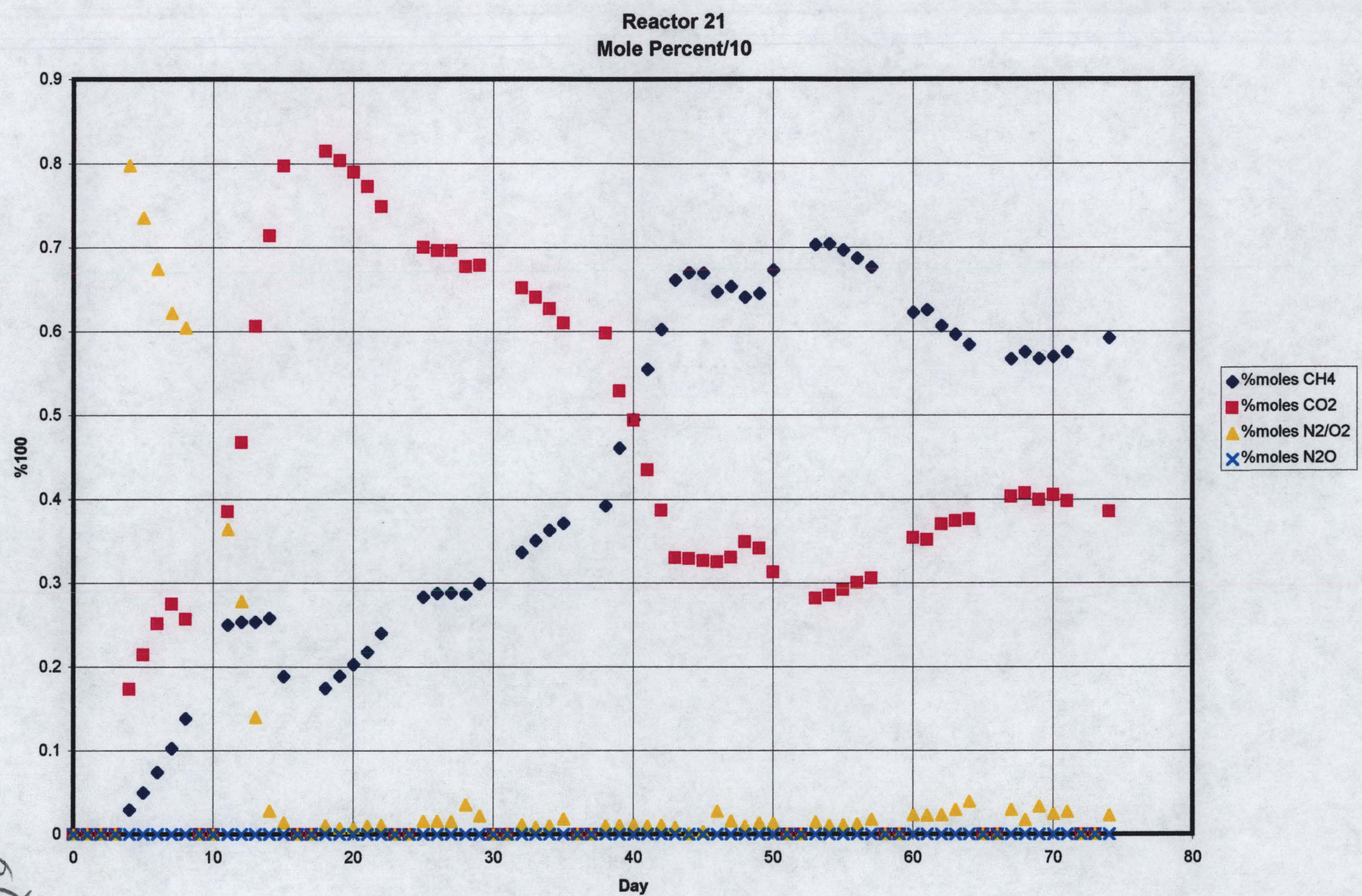
Reactor 19
Mole Percent/100
%100
Days
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1
0
10
20
30
40
50
60
70
80
%moles CH4
%moles CO2
%moles N2/O2
%moles N2O

36



Reactor 20
Mole Percent/100
%100
Day
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
10
20
30
40
50
60
70
80
%moles CH4
%moles CO2
%moles N2/O2
%moles N2O

69



Reactor 21
Mole Percent/10
%100
Day
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
0
10
20
30
40
50
60
70
80
%moles CH4
%moles CO2
%moles N2/O2
%moles N2O

98

Appendix 2

Constituent	Fly Ash	Green Liquor Dregs	Knots	Prim. Sludge	Sec. Sludge	Lime Mud
Macro-Nutrients (%)						
TKN	0.29	0.08	0.10	0.20	0.33	0.10
P	0.08	0.01	0.01	0.07	0.11	0.03
K	0.20	0.77	0.13	0.09	0.12	0.01
Ca	1.50	14.72	0.63	16.85	15.98	35.77
Mg	0.14	0.96	0.07	0.40	0.14	0.82
S	0.03	3.24	0.68	0.30	0.72	0.09
Micro-Nutrients (mg/kg)						
Fe	583.0	10,580.0	504.0	2,886.0	6,347.0	641.0
Mn	511.0	731.0	333.0	1,024.0	1,535.0	82.0
Zn	31.8	1,128.0	59.5	85.5	308.0	33.6
Cu	24.0	177.0	10.6	10.5	37.3	2.7
B	15.3	0.0	3.1	0.7	0.0	0.6
Heavy Metals (mg/kg)						
Ni	2.02	76.25	3.85	5.63	7.27	7.76
Cd	0.00	4.17	0.24	0.00	2.11	0.00
Pb	0.00	48.08	0.00	5.77	18.53	0.00
Other						
% Solids	21.23	45.92	18.92	26.23	36.36	61.04
Sodium (%)	0.02	12.25	2.14	0.36	0.36	0.51
pH	8.34	11.30	10.10	8.95	8.25	9.97
SAR	0.4	83.5	68.2	2.4	2.5	2.3
$CaCO_3$ eq. (%)	9.75	71.25	7.00	46.5	44.75	97.25

Table 1. NCDA Waste Analysis Laboratory Results (dry weight basis).

Waste Type	TKN (mg/kg)	NH_4-N (mg/kg)	NO_3-N (mg/kg)	TOC (mg/kg)	C/N Ratio
Flume Grits	236	15	1	396,000	1,671:1
Wood Yard Debris	1,110	4	2	293,000	263:1

Table 2. Aqua Tech analysis of flume grits and wood yard debris (dry weight basis).

Appendix 3a

Calculations to Determine Total Moles Gas Produced

I. Determine Mass Percent of Individual Gases Using GC

For example, CH_4=20%, N_2=20%, CO_2=60%

II. Calculate Mole Percent of Individual Gases

- Divide Mass Percent of Each Gas by Respective Molecular Weight

For example, $CH_4 = 0.02\ g / 16\ g\ mol^{-1} = 0.00125\ mol$

$N_2 = 0.02\ g / 28\ g\ mol^{-1} = .00071\ mol$

$CO_2 = .06 / 44\ g\ mol^{-1} = .00136\ mol$

- Sum Quotients From Above

For example, $0.00125 + 0.00071 + 0.00136 = 0.00332\ mol$

- Divide Mass Percent of One Gas by Its Molecular Weight, Divide by Quotient from Above, and Multiply by 100 to Determine Molar Percent

For example, $CH_4 = (0.00125\ mol / 0.00332\ mol) \times 100 = 37.6\%$

$N_2 = (0.00071\ mol / 0.00332\ mol) \times 100 = 21.4\%$

$CO_2 = (.00136\ mol / .00332\ mol) \times 100 = 41.0\%$

III. Determine Total Moles Gas Produced Using Formula n = PV/RT

- P = pressure generated in the reactor (in atm)

Pressure measured with U-tube of mercury, read as change in mmHg

Measured pressure change divided by 760 mmHg to standardize units (1 mmHg = 1 atm), and corrected for atmospheric pressure by adding 1 atm

For example, $\Delta mmHg = 20\ mmHg$

$P = 20mmHg/760mmHg\ atm^{-1} + 1\ atm = 1.026\ atm$

- V = volume of gas generated in the reactor (in L)

- Volume was calculated using the formula $V_1 = (P_2V_2)/P_1$, where V_1 = volume generated in the reactor (in L), P_2 = pressure generated in the reactor (in atm), V_2 = volume of reactor (in L) , and P_1 = 1 atm

- $V = V_1 - V_2$ + Change in volume in mercury tube

For example, $V_1 = (1.026\ atm \times 2.41\ L)/1\ atm = 2.472\ L$

- Add change in volume in mercury tube, which is $\pi r^2\ \Delta mmHg$

For example, $\pi \times (4mm)^2 \times 20\ mm = 1004.8\ mm^3 = .0010\ L$

$V = 2.472\ L - 2.41\ L + 0.0010\ L = 0.063\ L$

- R = Gas Constant = 0.08205 L atm deg^{-1} mol^{-1}
- T = Kelvin Temperature = 295 K
- Solve for n

For example, $n = (1.026\ atm \times 0.063\ L) / (0.08205\ L\ atm\ deg^{-1}\ mol^{-1} \times 295\ K)$

$n = 0.00267\ mol$

IV. Multiply Mole Percent of Individual Gas By Total Moles Gas Produced

For example, $CH_4 = 37.6\% \times 0.00267\ mol = 0.00100\ mol$

$N_2 = 21.4\% \times 0.00267\ mol = .00057\ mol$

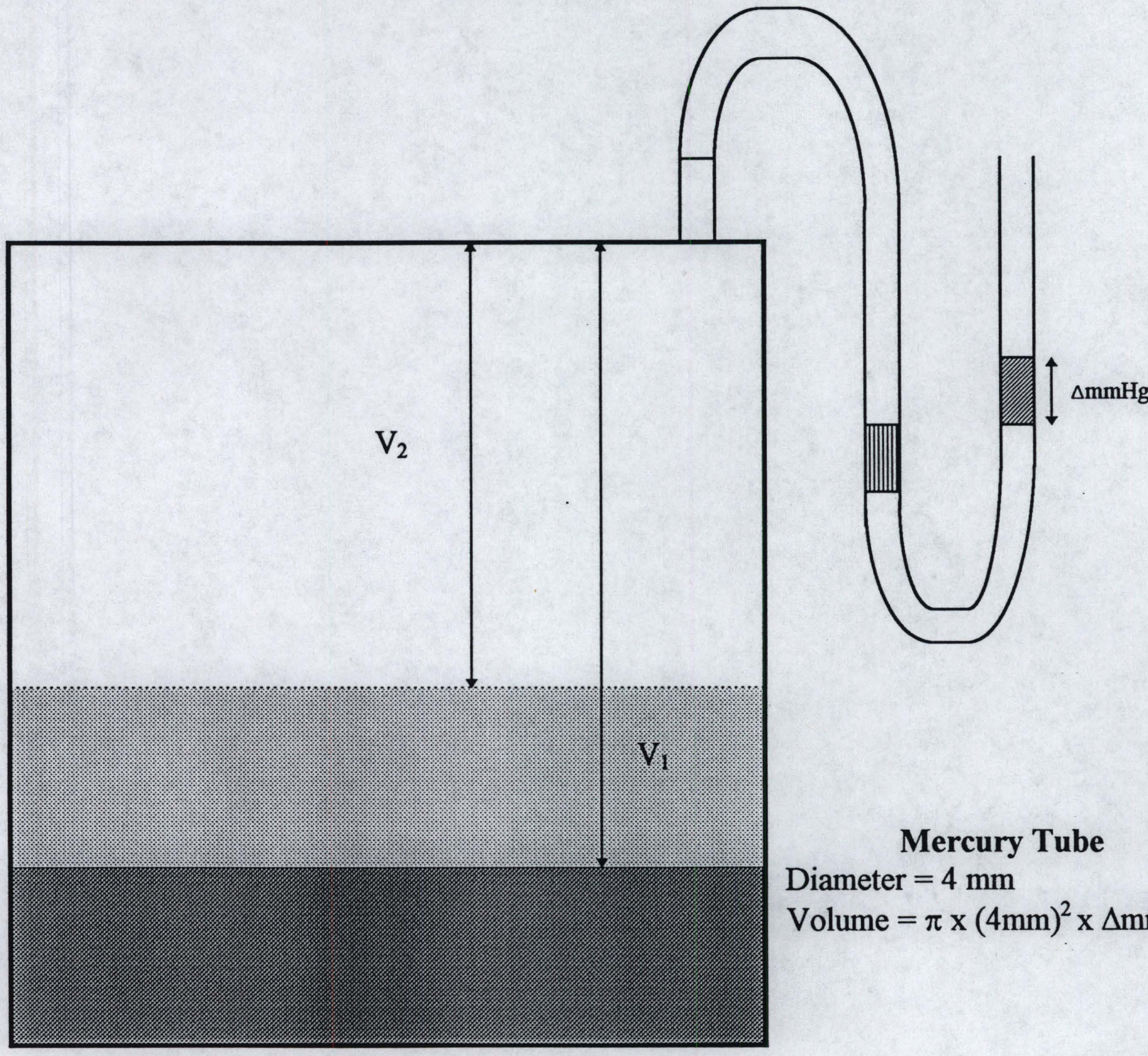
$CO_2 = 41.0\% \times 0.00267\ mol = .00109\ mol$

Appendix 22

Volume of Gas Generated in the Reactor

$$P_1V_1 = P_2V_2$$

$$V = V_1 - V_2 + \text{Volume Difference in Mercury Tube}$$



Mercury Tube
Diameter = 4 mm
Volume = π x $(4mm)^2$ x ΔmmHg

Reactor
Change in Volume Results from Increasing Pressure
V_2 = Volume under 1 atm pressure (measured constant)
V_1 = Volume under increased pressure
V = Total Gas Produced = $V_1 - V_2$